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Response surface illustration of interaction effects on CNMY2: growth temperature and time (p.51)

(a) Normal ECG signal(b) ECG signal at Apnea Episode (p.25)

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The authors declare no conflicts of interest.

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## SUSTAINABLE CONSTRUCTION IN OMAN: THE POTENTIAL ROLE OF THE INDUSTRIALIZED BUILDING SYSTEMS

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ABSTRACT: The Omani construction industry has been recently witnessing a boosted expansion associated with the notable growth in population and economy. In comparison with its neighboring countries, the construction industry in Oman is criticized for the lack of sustainability practices. Recently, the government and professional bodies have taken proactive actions to promote sustainable construction practices. The Industrialized Building Systems (IBS) could play an important role in the Omani construction industry in the future due to its environmental, economic, and social benefits. However, the lack of evidence on the contribution of IBS to the Omani construction industry suggests that this approach is not yet at the top of the stakeholder agenda. This paper focuses on the potential contribution of IBS to sustainable construction practices in Oman. Through a literature review, a comprehensive list of sustainability factors affecting IBS applications in the Omani construction industry was developed. A stakeholder survey of Omani construction industry including clients, engineers, and contractors was conducted to understand their perception on the importance of these factors. Statistical analysis revealed that the environment-related and the implementation-related factors are more important than the social-related factors. The economic-related factors were not found to be significantly more important than any other group of factors. Moreover, construction-time was found to be the most important and the most influential factor among the 62 factors considered in this study. The paper concludes with recommendations to promote and improve applications of industrialized building system in the Omani construction industry.

## الإنشاء المستدام في عمان: دور أنظمة الإنشاء المصنعة

م. ص. صـالح و ش. العلوش\*

الملخص: يشهد قطاع الإنشاء والتعمير في عمان نهضة كبيرة بسبب ازدياد أعداد السكان وتحسن الوضع الاقتصادي في العقود الأخيرة. إلا أن هذا القطاع ماز ال يتعرض للنقد بسبب انعدام التطبيق الكافي لمبادئ الإستدامة إذا ما قورن بالدول المحاورة. ونتيجة لهذا فإن كل من الحكومة والهيئات المهنية تسعى لاتخاذ إجراءات بهذا الخصوص. تشكل أنظمة الإنشاء المصاعرة في وضع الاستدامة في قطاع الإنشاء والتعمير العماني لما لها من فوائد بيئية واقتصادية واجتماعية. ونايشه في قطاع الإنشاء والتعمير العماني لما لها من فوائد بيئية واقتصادية واجتماعية. وبالرغم من هذا فإن كل من الحكومة والهيئات المهنية تسعى لاتخاذ إجراءات بهذا الخصوص. تشكل أنظمة الإنشاء والتعمير العماني لما لها من فوائد بيئية واقتصادية واجتماعية. وبالرغم من هذا فإن استخدام هذه الأنظمة لا يزال خجولا في عمان ويبدو انه ليس من أولويات الفئات المعنية بهذا القطاع. ولذلك، فإن هدف هذه الدراسة هو تحديد مدى مساهمة أنظمة الإنشاء المصنعة في دفع عجلة الاستدامة في عمان. اعتمدت الدراسة على طريقتين بحثيتين لتحقيق الهدف المرجو: أو لا, اعتمدت الدراسة على مراجعة المصادر العلمية المناع. المعدينة وذلك، فإن هم من هذا فإن استخدام هذه الدراسة هو تحديد مدى مساهمة أنظمة الإنشاء المصنعة في دفع عجلة الاستدامة في عمان. اعتمدت الدراسة على مراجعة المصادر العلمية المداسة ولالك، فإن هم رأي كل من المولي والموالين والموالين أنظمة الإنشاء المصنعة في معان. رائيا، استخدمت الدراسة المراسة على طريقتين بحثيتين لتحقيق الهدف المرجو: أو لا, اعتمدت الدراسة على مراجعة المصادر العلمية المناتيا استخدمت الدراسة التراسة المام لا يقم رأي كل من المهندسين والمقاولين وأصحاب المشاريع بخصوص أهمية هذه العوامل وأولوياتهم وذلك المتنيانا أستبيانا ألمام لغم رأي كل من المهندسين والمقاولين وأصحاب المشاريع بخصوص أهمية والعوامل وأولوياتهم في استبدانيا أولويات الموليا المعنية وذلك المتبيان ألمالا لقم رأي ي من المولي المام الإن الموليا وأولوياتهم وألمان البيئية والعوامل الراسة ولالي المنيان المنان ألمام أولول في عمان وأصحاب المشاريع بخصوص أهمية من العوامل الأخمة الإنسة والتبين أولويا وألما ألمالا المينية وستبين والمالين وأميمن الموامل وأولوياتهم في العوامل الأخرة ألمية من ألوقام وأولول الموام أي مالمر وألماناء الانساء والتعمير العماني الممامة ألى الدرا

*الكلمات المفتاحية:* قطاع الإنشاء والتعمير؛ أنظمة الإنشاء مسبقة الصنع؛ أنظمة الإنشاء المصنعة؛ عمان؛ الإنشاء المستدام؛ الاستدامة.

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Keywords: Construction industry; Industrialized building system (IBS); Oman; Sustainable construction; Sustainability.

## 1. INTRODUCTION

In spite of the cutbacks of public investments and the broad slowdown in the economy due to reduction in oil prices, the construction sector in Oman is still growing at a steady rate. In real terms, the construction projects registered in Oman were reported to grow by 9.4% annually during 2012–2016 (Timetric, 2017). The construction sector is the largest segment of the non-oil economy in Oman and as such, it remains a pillar in the government endeavor for diversification of the country's economy toward its long-term Vision 2040 (Oxford Business Group, 2018).

Achieving and maintaining sustainable development has become increasingly more prevalent for many countries over the last decades. In line with its neighboring countries, Oman has made plans for economic diversification to reduce its dependency on the fast depleting oil reserves, through building competencies across other sectors of the economy (The World Bank). Subsequently, the government had allocated huge investments on infrastructure, housing projects, and industrialization which had led to a tremendous boom across the construction industries (Oxford Business Group, 2013). This huge development is accompanied by the raising demand to maximize the efficient use of natural resources. The current global economic slowdown had put pressure on the Omani government to take steps to address issues related to quality, sustainability, and standardization in the construction sector to meet its needs and to bring its construction industry in par with international standards. The government of Oman has taken several actions to promote the development of sustainable construction practices. In spite of these efforts, many recent studies showed that the construction industry in Oman still lacks evidence of sustainability practices. Saleh and Alalouch (2015) have addressed several major challenges to the application of sustainable construction in Oman and grouped them into four major clusters: economic, capacity/professional, societal, and technological challenges. Similarly, Powmya and Abidin (2014) found that most of the stakeholders in Oman believe that applying sustainable practices in Oman would result in a dramatic raise in construction cost without any quantifying benefits, a view that is considered a challenge by itself. Moreover, Alalouch et al. (2016), in their comparative study of two energy-efficient houses in Oman and Qatar, concluded that applying sustainability practices in Oman still at early stages which require vast shifts in the construction industry. In a more recent study, the energy-saving potential in a residential building in Oman was explored. The result showed the potential for great saving in energy when energy passive measures are put in place (Alalouch et al., 2019). In fact, several recent research efforts have been focusing on the urban expansion in Oman (e.g. Alalouch et al. 2019; Alkamali et al.

2017).

On the other hand, the industrialized building systems can respond to many challenges in the construction sector in Oman. Zhang sees the IBS to be the future of the construction industry worldwide due to its ability for capitalizing on the strength of the industrial production (Zhang, Skitmore, and Peng, 2014). Industrialization from the construction point of view is considered part of the modernization process through developing methods of production and technology systems, where production operations are centralized, mechanized, and mass productionoriented (Lessing, 2006). Warszawski (1999) explained that an industrialization process is an investment in technology facilities and equipment, aiming at maximizing production output, minimizing demand on labor, and improving quality (Nawi et al., 2014).

Therefore, the objective of this study is to identify the sustainability criteria for IBS and assess their importance as perceived by the construction industry stakeholders in Oman. Recommendations that are proposed in this study could be used by different stakeholders to help to promote and improving the application of IBS in the Omani construction industry.

## 2. SUSTAINABILITY ASPECTS OF IBS

Though prefabrication and standardization terminologies are still in use, Industrialized Building System (IBS) has become the most common term used to represent those terminologies due to the research context of the Malaysian construction industry (Nawi, et al., 2014). Industrialized building construction is defined as a generic process of rationalization and standardization of work processes in the construction industry to reach cost efficiency, higher productivity and quality (CIB, 2010). A more elaborate definition for industrialized building construction is a change of thinking and practices to improve the production of construction and to produce high quality, custom-built, and environmentfriendly buildings through an integrated process, optimizing standardization, organization, cost, value, mechanization and automation (CIB, 2010).

The Industrialized Building Systems (IBS) is considered by several researchers to be the cornerstone rising for improving the construction industry, e.g. (Cook, 2005; Hampson and Brandon, 2004). This is due to the manufacturing of construction components in a controlled environment, either at-site off-site, then installed or assembled into or construction works. Although many studies have focused on the implementation of the IBS, there is a debate on its potential effect on the construction industry. For example, some studies considered that IBS is more expensive than conventional construction systems (Birkbeck and Scoones, 2005); while other studies found that project cost had been reduced remarkably due to IBS use over time (Goodier and

Perspective	Criteria				
	1. Reproduction, which ensures higher productivity and quality				
Economy	2. Simplified processes, which reduces the total energy involved				
	3. Working conditions to avoid losing time through severe weather				
Factory production	<ol> <li>Waste reduction due to modular coordination, bulk purchasing and factory applied finishes</li> </ol>				
	<ol> <li>Factory conditions, which avoid later repairs</li> <li>Precision in production keeping the</li> </ol>				
	construction site clean and free of debris				
	7. Flexible components, which allow for planning changes				
Adaptability	7. Demountable components, which allow for a major reconfiguration and relocation without demolition waste				

**Table 1.** IBS features that promote sustainability (Richard, 2006).

2007; Gibb and Isack, 2003). On the other hand, IBS has been linked to green construction (Jaillon and Poon, 2009; Zhang and Skitmore, 2012).

The sustainability aspects of implementing IBS practices can encourage the coordination between the built environment and construction processes, thus creating urban settlements that affirm healthy environments, social interaction, and encourage economic development. Richard identified eight key sustainability benefits of applying prefabrication in construction (Richard, 2006); as listed in Table 1. The following highlights the environmental, economic and social sustainability aspects of IBS in construction.

#### 2.1. Environmental Aspects

The environmental benefits derived from implementing IBS practices will vary according to the specific system implemented. With no doubt, typical traditional construction could have a better environmental performance over some prefabrication systems, but still the potential exists for IBS practices to have better environmental performance if they were well implemented and managed. As reported by Waskett (2001), one specific program being developed with the European-Commission backing has the potential anticipated benefits associated with IBS of a 50% reduction in the amount of water used for the construction of a typical house, and a 50% reduction in the use of quarried materials used in the construction. Another environmental benefit of using IBS elements is that conventional formworks could be eliminated or greatly reduced, which will reduce the issue of construction waste and its subsequent environmental problems. Prefabrication can also promote a safer working environment for construction workers. The need of a large number of workers and raw materials are also noticeably reduced at the sites. In addition, as products are manufactured in a controlled environment and standard in sizes, wastage will be minimized at both factory and construction sites (Bari, *et al.*, 2012).

Equally important, IBS has the potential to accommodate modern and smart building energy systems (mechanical, electrical, fire safety, etc.) that are known for their high environmental performance as IBS has been seen as the future of the construction industry (Zhang, Skitmore, and Peng, 2014) and linked to green construction (Jaillon and Poon, 2009).

#### 2.2. Economic Aspects

There are many economic benefits for implementing IBS practices in construction; among them are cost savings, quality, and speed of construction. Operation and maintenance costs are reduced due to the highquality characteristics of prefabricated components. Yunus and Yang (2011) mentioned that the cost of labor and materials will be also reduced significantly by adopting IBS. On the other hand, Kamaruddin et al. (2013) revealed that a considerable number of contractors are unwilling to take part in projects that include IBS. This is due to the higher costs associated with the procurement of materials and hiring welltrained labour despite the overall cost saving it offers. Jaillon and Poon (2009) concluded that adopting IBS practices in a project could lead to an overall reduction of 16% of labour costs, as well as 15% of total construction time. In addition, Goodier and Gibb (2007) and Ding (2008) emphasized that due to its standardization process; IBS will result in a significant time saving, improve quality control, and reduce material. To achieve these benefits all of the construction team must realize and understand the sustainability potential of IBS. This review demonstrates the sustainability potential of IBS as one of the modern construction systems that could achieve better economic viability if it is properly utilized and implemented.



Figure 1. Potential reduction in cost when industrialized construction is used (CIB, 2010).

#### 2.3. Social Aspects

Adopting prefabrication in construction will have a remarkable effect on the labour market. Investing in automation will help in reducing the dependence on unskilled workers and foreign labours (El-Abid and Ghazalia, 2015). Prefabrication can also promote healthier working environments as a result of cleaner and safer construction sites. In addition, it improves the health of the building's occupants by controlling moisture and volatile organic compounds levels in a factory setting. Controlling the quality of building components will lead to minimizing risks of chronic health issues of occupants (Ghaffarianhoseini, et al. 2018). In urban areas, traffic disruption to congested roadways due to construction activities is very crucial. Traditional construction practices usually result in pollution, noise, dust, and require more trips to the construction site. On the contrary, IBS can reduce onsite construction activities and time, which inevitably reduce the disturbance associated with construction. In addition, prefabricated units are normally transported in large sizes leading to a limited number of trips to the site, and thus creating less disruption to the congested city traffic and the local community.

## 3. DEVELOPMENT OF A COMPREH-ENSIVE LIST OF SUSTAINABILITY FACTORS FOR IBS APPLICATION

A literature review was conducted aiming at developing a comprehensive list of typical sustainability factors affecting the quality of IBS implementation. Although the implementation of IBS practices vary from a country to another, some applications reflect the adoption of IBS in developing countries like Oman. Among these applications are 'Standardization' (Gann, 1996; Lessing, Stehn, and Ekholm, 2005; Pasquire, Gibb, and Blismas, 2004), 'Prefabrication' (Dawood, 1996; Song, 2005) and 'System Building' (Finnimore, 1989; Luo, Riley, Horman, and Kremer, 2008). Sustainability factors related to these applications were reviewed, summarized and categorized according to the main pillars of sustainability, which are economy, environmental, and social, in addition to implementation-related factors. It is worth mentioning that each factor could have been broken further into "indictors", but this would have resulted in an unmanageable number of variables. Equally important, this study focuses on identifying and evaluating the overarching factors that might facilitate or hinder the implementation of IBS in Oman. Hence, the literature review was limited to the factors only as summarized in Table 2.

 Table 2. Sustainability factors for IBS application.

ECONOMIC-RELATED FACTORS					
Criteria	Code	Factors			
Cost	Ec-01	Maintenance and operation costs			
	Ec-02	Disposal costs			
	Ec-03	Life cycle costs			
	Ec-04	Initial construction costs			
	Ec-05	Material costs			
Time	Ec-00 Ec-07	Construction time			
Time	Ec-08	Lead-times			
	Ec-09	Speed of return on investment			
	Ec-10	Transportation and lifting			
	Ec-11	Production			
	Ec-12	Design stage adoption			
SOCIAL-REI	LATED FA	CTORS			
Criteria	Code	Factors			
Social Issues	Sc-01	Local economy			
	Sc-02	Participation and control			
	Sc-03	Community disturbance			
	Sc-04	Traffic congestion			
	Sc-05	Public awareness			
	Sc-06	Public participation			
Labour	Sc-07	runciples and values			
Market	Sc-00	Knowledge and skills			
	Sc-10	Labor availability			
Safety and	Sc-11	Workers' health and safety			
Health	Sc-12	Working conditions			
	Sc-13	Disaster preparedness			
Design and	Sc-14	Site attributes			
Arch issues	Sc-15	Aesthetic options			
ENVIRONMI	ENT-RELA	ATED FACTORS			
Criteria	Code	Factors			
Waste	En-01	Waste generation			
Г	En-02	Waste disposal			
Consumption	En-03 En-04	Design and construction			
Consumption	En-05	Operational energy			
Recycle	En-06	Recyclable / renewable contents			
2	En-07	Reusable / recyclable elements			
Pollution	En-08	Site disruption			
	En-09	Pollution generation			
	En-10	Environment administration			
	En-11 En 12	Ecology preservation			
	En-12 En-13	Inclusive environment			
Resource	En-13 En-14	Water consumption			
Consumption	En-15	Land use			
	En-16	Material consumption			
IMPLEMENT	TATION-R	ELATED FACTORS			
Criteria	Code	Factors			
Technical	Tc-01	Durability			
Quality	Tc-02	Detects and damages			
	Tc-03	Loading capacity			
	1c-04	Integration of building services			
	10-05 Te 06	Constructability			
	Tc 07	Usage efficiency			
	Tc-07	Adaptability and flexibility			
	Tc-09	Technology			
Enforcement	Im-01	Standardization			
and	Im-02	Governance			
Regulations	Im-03	Legislation			
	Im-04	Policy and strategy match			
	Im-05	Building capacity			
	Im-06	Design standard and project function			
	Im-07	Project control guidelines			
	Im-08	Integrated environmental and			
		economic program			
	Im-09	Procurement system			

## 4. DATA COLLECTION AND ANALYSIS

#### 4.1. Survey Design and Sample Characteristics

The survey aimed to investigate the perspective of the Omani construction industry's stakeholders on the relative importance of sustainability factors for IBS applications in Oman. A pilot study was conducted to ensure the effectiveness of the survey; accordingly, it was developed and distributed to the target groups.

The questionnaire survey consisted of three parts. The first part focused on collecting background information about the respondent including years of experience in the construction industry, profession, organization, and the number of IBS projects. In the second part, respondents were asked to rate the importance of each sustainability factor; presented in Table 2; with respect to the IBS applications. A fivestep Likert-scale was used, in which (1) refers to "least significant" and (5) "most significant". To ensure consistency in understanding the factors across participants, the questionnaire included a clarifying definition for each factor. In the third part, respondents were given the opportunity to provide supplementary factors in an open-ended question.

A total of 80 questionnaires were distributed and only 54 responses were received, which gives an overall response rate of 67.5%. Among those 54 respondents, 19% were from clients/developer sub-group, 43% from consultants/designers, 19% from governmental institutes, and 13% from contractors. Furthermore, 50% of the respondents were practicing as engineers, 26% architects, and 15% project managers. All of the survey participants had professional experience with industrialized building systems. About 52% had an experience of less than 5 years, while 48% had more than 5 years of IBS experience. Fig. 2 shows a detailed distribution of respondents among different sub-groups.

#### 4.2. Data Analysis and Discussion

In order to identify the most important factors as perceived by the respondents, the data was analyzed statistically. For the purpose of this study, descriptive analysis and tests of differences of the important data of the factors considered are reported.

# 4.2.1. The importance of the categories of factors

The first question in this study was to identify the most important group of factors as perceived by the respondents. Hence, the mean score for the factors in each one of the four categories was calculated across all responses. Fig. 3 shows that the Environment-related factors are seen as the most important group of factors followed by the implementation-related factors. The least important group was the social-related factors whereas the Economic-related factors came in the third place.









able 5. Normanty of the variables.								
	Economic- related	Environment- related	Social-related	Implementati on-related				
Kolmogorov- Smirnov	0.96	0.11	0.109	0.109				
Sig.	0.2	0.1	0.16	0.16				

Paired-sample t-test	Economic-related	Environment-related	Social-related	Implementation-related
Economic-related	0			
<b>Environment-related</b>	-1.47 (0.148)	0		
Social-related	0.63 (0.53)	3.1 (0.00)*	0	
Implementation-related	-1.1(0.28)	0.43 (0.67)	-2.39 (0.02)**	0
* Significant at 0.01. ** Signif	ficant at 0.05			

Table 4. Differences between the variables - t (sig.).



Figure 4. Overall importance of the IBS sustainability factors based on the ranking of these factors.



Figure 5. The influence of IBS sustainabililty factors. Each data point represents one factor and the associate number represents the factor's overall rank.

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Table	5 Statistical	analysis and	ranking (	of sustainability	v factors for	IBS and	lications in	Oman
I aDIC	J. Statistical	analysis and	Tanking (	or sustamaonne	$1a_{10}$	IDS abl	meanons m	Oman.

Table 5. c	Statistical analysis and fanking of sustaina		Do applications	III OIIIaII.	
SUSTA	INABILITY PERFORMANCE	Mean Value*	Standard	Overall Importor of the	Overall
FACIO	VKS MIC DELATED EACTODS		Deviation	Importance^^	Influence ***
ECONO Ec-07	Construction time	4 11	1.09	Н	01
Ec-07 Ec-02	Disposal costs	3.78	1.16	M-H	Q2
Ec-11	Production	3.57	1.25	Μ	Q4
Ec-08	Lead-times	3.56	1.00	М	Q3
Ec-09	Speed of return on investment	3.56	1.18	M	Q4
EC-U3 Ec 12	Life cycle costs	3.50	1.11	M M	Q3 Q3
Ec-12 Ec-01	Maintenance and operation costs	3.30	1.00	M-L	Q3 04
Ec-04	Initial construction costs	3.37	1.26	M-L	Q4
Ec-05	Material costs	3.37	1.19	M-L	Q4
Ec-06	Labour cost	3.35	1.26	M-L	Q4
Ec-10	Transportation and lifting	3.	1.00	L	Q3
ENVIR	ONMENT-RELATED FACTORS	<b>a</b> o <b>z</b>	1.0.5		
En-01 En 00	Waste generation	3.87	1.05	M-H M H	OI O1
En-09 En-02	Waste disposal	3.83	1.02	M-H	01
En 02 En-10	Environment administration	3.80	1.05	M-H	01
En-07	Reusable / recyclable elements	3.76	1.10	M-H	Q1
En-12	Health of occupants (indoor air	3.76	1.26	M-H	Q2
En-14	Water consumption	3.67	1.30	М	Q2
En-04	Energy consumption in design and	3.65	1.08	M	Q1
En-15	Land use	3.61	1.14	M	Q2
En-06 En 11	Recyclable / renewable contents	3.5/	1.13	M	Q4 Q4
En-11 En-08	Site disruption	3.37	1.14	M	03
En-05	Operational energy	3.46	1.18	M	04
En-16	Material consumption	3.46	1.14	М	Q4
En-03	Embodied energy	3.39	1.19	M-L	Q4
En-13	Inclusive environment	3.37	1.15	M-L	Q4
SOCIAI	L-RELATED FACTORS				
Sc-11	Workers' health and safety	3.89	1.19	M-H	Q2
Sc-03 Sc-12	Working conditions	3.80	1.10	М-н М-н	$Q^2$
Sc-09	Knowledge and skills	3.72	1.11	M	01
Sc-13	Disaster preparedness	3.70	1.09	M	01
Sc-05	Public awareness	3.59	1.04	Μ	Q3
Sc-10	Labor availability	3.59	1.17	М	Q4
Sc-14	Site attributes	3.54	1.06	M	Q3
Sc-01	Local economy	3.43	1.13	M-L M I	Q4 Q2
Sc-04 Sc-06	Public participation	3.33	1.05	M-I	Q3 04
Sc-16	Physical space	3.28	1.12	M-L	04
Sc-15	Aesthetic options	3.20	1.14	L	Q4
Sc-07	Principles and values	3.15	1.16	L	Q4
Sc-08	Influence on job market	3.15	1.22	L	Q4
<u>Sc-02</u>	Participation and control	3.11	1.11	L	Q3
TMPLE	MENTATION-RELATED FACTORS	4.00	1.12		02
1 c-02 Tc-05	Defects and damages Integration of supply chains	4.00	1.13	H M-H	Q2 01
Tc-05	Constructability	3.83	1.15	M-H	$\frac{Q^1}{O2}$
Im-01	Standardization	3.76	1.23	M-H	Q2
Tc-01	Durability	3.72	1.05	М	Q1
Tc-03	Loading capacity	3.72	0.93	М	Q1
Tc-07	Usage efficiency	3.63	1.16	M	Q2
1m-05 Im-04	Building capacity	3.01 3.50	1.08	M M	Q3 Q3
Te-04	Integration of building services	3.59	1.00	M	03
Im-07	Project control guidelines	3.57	1.00	M	03
Im-03	Legislation	3.54	1.07	M	Q3
Im-08	Integrated environmental and	3.54	1.11	М	Q3
Im-04	Policy and strategy match	3.50	1.04	М	Q3
Im-09	Procurement system	3.46	1.23	M	Q4
1m-02	Governance	3.3/ 2.21	0.94	M-L M-I	Q3
1 C-Uð Te-00	Adaptaoliny and nexibility	5.51 3.30	1.24 1.10	M-L	Q4 04

\* The order of the factors reposes the level of importance within the group. \*\* Based on Fig. 4. H= high, M-H= medium-high, M= medium, M-L= medium-low, L= low. \*\*\* Based on Fig. 5. Q1= High importance, high consensus, Q2= High importance, Low consensus, Q3= Low Importance, high consensus, Q4= Low importance, Low consensus.

However, the figure shows that the differences between the groups are marginal. It was necessary therefore to test the statistical significance between the differences in the mean scores of the groups. Therefore, the normality of the data was examined using the One-sample Kolmogorov-Smirnov test (K-S test). The significant value of this test (p < 0.05) indicates a deviation from normality. The results of this test indicated that all variables are normally distributed as shown in Table 3. Hence, the pairedsample t-test was used to check if the differences between the importance of the variables are statically significant.

The results of the test of differences is reported in

Table 4, which shows that the Environment-related factors were seen as significantly more important than the social-related factors (t=3.1, df= 53, p < 0.01). Whereas, the participants valued the Implementation-related factors significantly more than the social-related factors (t= -2.39, df= 53, p < 0.05). This suggests that the Environment-related and the Implementation-related factors are more important than the social-related factors. In contrast, no significant differences were found between the economic-related factors and any other variable.

#### 4.2.2. The overall importance of the factors

The next step was to identify the most important factors among the 62 factors considered in this study. Therefore, the average score for each factor was calculated as shown in Fig. 4. A close examination of the trend line revealed a clear break in the mean score at four points, which suggests that the factors can be split into five levels in terms of importance. The level with the highest important factors contains only two factors namely, Construction Time and Defects and Damages. On the other hand, the low importance level has five factors i.e. Transportation and Lifting, Aesthetic Options, Principles and Values, Influence on Job Market, Participation and Control.

Although Fig. 4 provides an insight into the structure of the importance of each factor based on a simple ranking of the mean score, it does not take into account information regarding the consensus among the participants' answers. In order to explore the relationship between the level of importance and the level of consensus, the mean score and the standard deviation were normalized to render them comparable. Fig. 5 shows a scatter plot between the two normalized variables. In this figure, the average of each axis was used to split the factors into four quarters based on their level of Influence (i.e. importance and consensus). The results show that there is a general consensus about twelve important factors out of the 62 factors included in this study which are shown in the figure in the Q1 quarter (Q1= high mean value with low St. deviation). These factors are: 1-Construction time, 4-Waste generation, 5-Pollution 6-Waste disposal, 7generation, Integration of supply chains, 9-Environment administration, 13-Reusable/recyclable elements, 16-Knowledge and skills, 17-Durability, 18-Loading capacity, 19-Disaster preparedness, 21-Energy consumption in design and construction. This suggests that these factors have the highest potential to influence the adoption of IBS in the Omani market.

Table 5 shows the mean score, standard deviation, importance level and influence level for each factor. Construction Time was considered the most important and the most influential factor across all factors. In the Environment-related factors attention should be given to Waste Generation, Pollution Generation, Waste Disposal, Environment administration, and Reusable/recyclable elements. As for the socialrelated factors, Workers' Health and Safety, Community Disturbance and Working Conditions were seen as the most important and most influential factors in this group. The results for the Implementation-related factors are not inclusive.

## 5. CONCLUSION

This study presented the sustainability factors affecting the implementation of IBS in the context of Oman construction industry. The research identified 62 factors based on the main pillars of sustainable development, consisting of 12 economy-related factors, 16 environment-related factors, 16 socialrelated factors, and 18 implementation-related factors. A questionnaire was conducted to investigate the perspective of the Omani construction industry stakeholders on the relative importance of these factors. Analysis of the collected data revealed that environment-related factors were generally considered the most important factors related to IBS, followed by implementation-related factors and social-related factors, while economic-related factors were the least important. The top three environment-related factors included En-01 (Waste generation), En-09 (Pollution generation), and En-02 (Waste disposal). The top three implementation-related factors include Tc-02 (Defects and damages), Tc-05 (Integration of supply chains), and Tc-06 (Constructability). The top three social-related factors include Sc-11 (Workers' health and safety), Sc-03 (Community disturbance), and Sc-12 (Working conditions). Finally, the top three economic-related factors include Ec-07 (Construction time), Ec-02 (Disposal costs), and Ec-11 (Production). Equally important, Construction Time was found to be the most important factor and the one that has the highest consistency level concerning its importance.

The Omani government and construction professionals can use the findings of this study as guidelines to focus their effort in promoting the application of IBS in Oman by emphasizing the factors that matter the most to the stakeholders while fitting within the government's short- and long-term strategy and targets. On the other hand, the study provides essential information to the decision-makers in the construction industry to understand the key areas where the implementation of IBS practices needs improvement, leading to implement proper actions and management strategies to maximize the benefits of IBS practices. In addition, the findings of this study lay the groundwork for future investigation of effective actions to manage these sustainability factors in the Omani construction industry. Future research in this area should focus on developing measurable "indicators" for each factor that are meaningful for the Omani construction industry and explore the differences in the importance of these factors across building types.

#### **CONFLICT OF INTEREST**

The authors declare no conflicts of interest.

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## APPLICABILITY OF USING REVERSE OSMOSIS MEMBRANE TECHNOLOGY FOR WASTEWATER RECLAMATION IN THE GAZA STRIP

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**ABSTRACT:** Reverse osmosis (RO) technology shows common popularity in the field of water treatment as an advanced stage to eliminate the residual biogenic elements and dissolved impurities after the traditional treatment processes. This article highlights the applicability of using RO membrane technology as a post-treatment stage to treat the discharged effluent from the Gaza wastewater treatment plant. The designed experimental model reveals optimal removal efficiency between 92 and 100% for a number of physical, chemical and biological pollutants. The RO membrane unit demonstrates significant removal efficiency compared to the sand filter where the RO removal efficiency for BOD, TSS, TDS, Fecal Coliform and NO<sub>3</sub> were 100, 97.5, 92, 100 and 100%, respectively. The quality of reclaimed wastewater was idealistic where the contents of BOD, Fecal Coliform and NO<sub>3</sub> in the permeate were nil, and the concentrations of TDS and TSS were 20 and 296 ppm, respectively. Practically, the results confirm that the wastewater with the reclaimed quality could be used for agricultural activities with no degree of restriction according to FAO's guidelines water quality for irrigation. According to the Palestinian Standard (PS), the quality of reclaimed wastewater is high, class (A), and it could be used without restrictions to irrigate many crops and for the purposes of groundwater replenishment. Related to energy estimation and cost analysis, the numerical model and the market analysis study demonstrate the energy of 1.23 kWh and total cost, i.e. fixed and energy costs, of 0.58 USD to produce 1 m<sup>3</sup> of reclaimed wastewater using the RO membrane in the Gaza Strip over a projected lifespan of 5 years.

Keywords: Gaza Strip; Wastewater; Reclamation; Reverse osmosis; Post-treatment; Agriculture.

## المياه العادمة في قطاع غزة مدى قابلية تطبيق تقنية التناضح العكسي لاستصلاح

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الملخص: في ضوء التطور السريع في تكنولوجيا معالجة المياه، أصبح توجه استصلاح المياه العادمة خياراً واعداً لتخفيف أزمة المياه ولزيادة الاستدامة في نظام إدارة المياه في المناطق التي تعاني عجز حقيقي في مصادر المياه. تكنولوجيا التناضح العكسي تشكل التقنية الأكثر رواجاً في مجال معالجة المياه و إز الة العوالق و الملوثات البيولوجية و غيرها. نسلط الضوء في هذا المقال على استخدام أغشية التناضح العكسي لمعالجة المياه العادمة الخارجة من محطة غزة للمياه العادمة و ذلك للوصول إلى مياه قابلة للاستخدام في مجال الزراعة و حقن الخزان الجوفي حسب المعايير الفلسطينية للمياه. أظهر النموذج المصمم باستخدام أغشية التناضح العكسي كفاءة تتراوح بين 92 إلى 100% في إز الة الملوثات البيولوجية و فير ها. والبيولوجية. وكانت كفاءة إز الة لـ BOG، STSS (BOD، 2016) في إز الة الملوثات الفيزيائية ،الكيميائية والبيولوجية. وكانت كفاءة إز الة لـ MOG، STSS، BOD، في إز الة الملوثات الفيزيائية ، الكيميائية والبيولوجية. وهذا تعني أنه يمكن الاستنتاج أن جودة المياه المعالجة بهذه التقنية يمكن تصنيفها بدرجة (A) حسب المعايير الفلسطينية، وهذا تعني أنه يمكن الاستنتاج أن جودة المياه المعالجة بهذه التقنية يمكن تصنيفها بدرجة (A) حسب المعاير الفلسطينية، وهذا تعني أنه يمكن الاستنتاج أن جودة المياه المعالجة بهذه التقنية يمكن تصنيفها بدرجة (A) حسب المعاير والبيولوجية. وحانت كفاءة اللازمة يومياً هي 102، 2015 علمان مالموثات الفيزيائية ، الكيميائية والبيولوجية. وكانت كفاءة إز الة الموثان المعالجة بهذه التقنية يمكن تصنيفها بدرجة (A) حسب المعاير والبيولوجية. وهذا تعني أنه يمكن الاستنتاج أن جودة المياه المعالجة بهذه التقنية يمكن تصنيفها بدرجة (A) حسب المعاير والولار الموليزيزيزيز مالخان الجوفي. من ناحية عرفي وحقن الخزان الجوفي. من ناحية أخرى

الكلمات المفتاحية: قطاع غزة؛ مياه عادمة؛ استصلاح؛ تناضح عكسى؛ ما بعد المعالجة؛ زراعة.

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### 1. INTRODUCTION

The water scarcity crisis has raised as one of the most pressing problems on the sustainability of life due to the effect of population growth, climate change, water abuse, pollution of water resources, improper management of water and other factors (Mehta, 2007; Karagiannis and Soldatos, 2008; Vörösmarty et al. 2010; Jacobson et al. 2011 Ang et al. 2015). The water crisis threatens about four out of every five persons of the human communities and more than two-thirds of the environmental habitats (Jacobson et al. 2010; Vörösmarty et al. 2010). In an effort to alleviate the water shortage, new water sources have been identified and supplied through desalination of seawater or brackish water, and by reclaiming wastewater (Zhang and He, 2013). However, the utilizing of treated wastewater in agriculture and in the replenishment of water resources (i.e., surface and groundwater) is less expensive than using the desalinated water and it is more affordable for most crops (FAO 2006; Zhang and He, 2013; Jaramillo and Restrepo 2017). Moreover, the worth of wastewater comes from being a promising constituent in promoting smart sustainable development especially in arid and semi-arid areas and many counties worldwide. The merit of wastewater as an efficient alternative water source is derived from the fact that about 99% of its ingredients are water and only less than 1% is solid wastes and hence the waste components reduction of wastewater to acceptable levels by means of water recycling could be worthy environmentally and economically to the sustainability of water resource management.

Nowadays, the rapid development in membrane manufacturing technologies, such as micro, ultra, nanofiltration, and reverse osmosis (RO) membrane technologies plays a significant role in enhancing the feasibility of water and wastewater reclamation by adding a quaternary-post-treatment process to remove the remaining pollutants, e.g. pyrogens, color, submicron colloidal matter, bacteria, and viruses, after the tertiary treatment (Metcalf and Eddy, 2013). In comparison to other membrane technologies, the use of RO technology is the most prestigious in the purification of wastewater due to the low energy consumption and the high rate of pollutant and contaminant removal. Edwards and Schubert (1974) and Fang and Chian (1976) reported that the RO membrane technology could eliminate 51% up to 99% of various pesticides. Al Jlil and Sajid (2014) found that the RO water treatment process could remove all the cations and anions from wastewater or seawater especially removing the monovalent ions such as chloride (Cl<sup>-</sup>) with a removing efficiency of 94.4%. Suzuki and Minami (1991) revealed through several experiments that RO membranes could remove up to 99%, 90% and 99.9% of total dissolved solids (TDS), total organic carbon (TOC) and fecal coliform, respectively. Abid et al. (2012) addressed the dye removal efficiency of more than 97% from industrial wastewater by membrane technologies of RO.

Practically, agriculture shows a huge demand for water as it is considered the biggest consumer of water in volume among all other water sectors. The possibility of using reclaimed wastewater forms an attractive water source to farmers for several reasons such as its reliability, its low or zero cost and its valuable nutrient content, which increases crop production without adding artificial fertilizers (U.S. EPA, 2004; Jiménez, 2005; Qadir et al. 2010). In specific, the typical domestic wastewater is a rice source of the nitrogen, phosphorus, potassium, micronutrients and organic matters that are naturally required for agricultural harvesting (FAO 1992). However, the main challenge that faces the sustainability of any wastewater reclamation activity is compromised by public health assurances. The wastewater is most likely to contain pathogenic organisms similar to those in the original human excreta such wastes: bacteria, viruses, protozoa, and helminth (FAO 1992). Depending on the harsh effects of sunlight and desiccation, almost all excreted pathogens can survive in soil and on crop surfaces for a sufficient length of time ranges between two days and many months which is a sufficient to indicate that public health risk and infections by Diarrhea, Typhoid, Salmonella type and other virus diseases could be conjugated (WHO 1989). Moreover, the accumulation of heavy metals the wastewater demonstrates a major potential risk to consumers and farmers (Jaradat, 2016). Gumbo et al. (2010) found significant public health risks for using wastewater for agricultural irrigation. Jan et al. (2010) detected heavy metals concentrations in a number of tested food crop samples irrigated with wastewater. Generally, the long term using of partially treated wastewater in irrigation can degrade soil and cause public health risks through the transmission of enteric diseases and heavy metal accumulation (Jamil et al. 2010). Obviously, the different water activities and uses in terms of public health and environmental protections are principally governed by specific guidelines and frameworks that are set and adopted by the World Health Organization (WHO) or by the national legislation of countries. In Palestine, the relevant policies, legislation, and regulation related to water quality are set and adopted by the Palestinian Water Authority (PWA) with direct negotiations and discussion with the related ministries such as the ministry of health, ministry of agriculture and the environmental quality affairs (PWL, 2002). Generally, the wastewater treatment passes through a tertiary treatment process which contains an Ultraviolet (UV) disinfection stage to eliminate the pathogenic organisms from the treated wastewater. Unfortunately, the technique of UV is unavailable in most of the regions that suffer from a lack of facilities and energy resources. The wastewater is a valuable source of water to balance the water deficit in the Gaza Strip, therefore, this study demonstrates the applicability of using the RO membrane technology as a post-treatment stage instead of the UV stage to eliminate the remained nutrients and pathogenic organisms from the reclaimed wastewater in order to be efficient in term of quality to be applicable in different water sectors according to specified Palestinian water standards and guidelines. Moreover, not only in the Gaza Strip, the applicability of RO membrane technology as a posttreatment stage could be a potential unique solution for different countries in the Middle East and North Africa

which suffer from poor facilities and energy resources to overcome the need to tertiary treatment stage of UV which needs a well-established preparations and facilities.

## 2. ENVIRONMENTAL STANDARDS AND REGULATIONS

Investigating the efficiency of any water treatment systems is performed in terms of the produced quality of water and its coinciding with the adopted guidelines and standards. Water quality, which is as significant and important as quantity, has been overlooked for decades in terms of legislation, investment and public awareness. Water and sanitation infrastructure, water and sanitation policies, good governance and practice, proper legislation, regulations, and standards are key issues to safeguard humans and their surrounding environment. Various legislations and regulations were designed worldwide to regulate water resource protection, water supply, sanitation, protection of the environment, and prevention of pollution. Standards have been adopted for different purposes of water uses and reuse. Monitoring systems have been put in place to ensure responding to these regulations and standards. The availability of adopted water quality standards and regulatory aspects in forms of national or international standards is important to control and restrict the arbitrary use or disposal of wastewater. There are no unified criteria for wastewater reuse in the world and each country proposes its own best practices criteria based on different environmental, economic, social and political conditions. Globally, many organizations and agencies like the World Health Organization (WHO), Food and Agriculture Organization (FAO) and Environmental Protection Agencies (EPAs) suggest guidance standards for wastewater reuse for different purposes. In the 1970s, the WHO started developing guidelines, shown in Table 1, for wastewater reuse in agricultural irrigation to ensure human health and environmental integrity. These guidelines are based on the consensus view that the actual risk associated with irrigation with treated wastewater is much lower than previously thought and that earlier standards and guidelines for effluent quality (WHO, 2006). Traditionally, irrigation water is grouped into various quality classes in order to guide the user to the potential advantages as well as problems associated with its use and to achieve optimum crop production. The water quality classifications are only indicative guidelines and their application has to be adjusted to conditions that prevail in the field. This is so because the conditions of water use in irrigation are very complex and difficult to predict (WHO, 2006). The suitability of water for irrigation greatly depends on the climatic conditions, physical and chemical properties of the soil, the salt tolerance of the crop grown and the management practices. Thus, the classification of water for irrigation will always be general in nature and applicable under average use conditions. Many schemes of classification for irrigation water have been proposed. FAO (1985) classified irrigation water into three groups based on salinity, sodicity, toxicity, and miscellaneous hazards. These general water quality classification guidelines help to identify potential crop production problems associated with the use of conventional water sources. The guidelines are equally applicable to evaluate wastewaters for irrigation purposes in terms of their chemical constituents, such as dissolved salts, relative sodium content and toxic ions. Depending on the sodium ion concentration relative to the concentrations of calcium and magnesium ions (as indicated by SAR) and the total salt concentration, the potential content of sodium ions in the irrigation water affects the infiltration rate and the soil permeability. Obviously, it is clear to indicate that for a given SAR value, an increase in total salt concentration is likely to increase soil permeability and, for a given total salt concentration, an increase in SAR will decrease soil permeability. This illustrates the fact that soil permeability (including infiltration rate and surface crusting) hazards caused by sodium in irrigation water cannot be predicted independently of the dissolved salt content of the irrigation water or that of the surface layer of the soil (FAO 1985). In Palestine, the Palestinian water laws clearly declare the responsibility of the PWA, in consultation with the ministry of health, ministry of agriculture and environmental quality authority, to establish guidelines and framework for monitoring the water-related practices, protecting the water resources and ensuring the safety of Palestinian public health (PWL 2002, 2014). The Palestinian Standard (PS 2003) imposes a set of mandatory technical instructions for using treated wastewater in agricultural irrigation to maintain the safety of humans, animal health and the crops as well as to sustain environmental constituents. The PS prohibits the usage of treated wastewater for watering livestock and poultry, irrigation of all types of vegetables, groundwater recharge through direct injection and fish farming. Based on the quality of treated wastewater, the PS categorizes the effluents into four classes based on the water quality as shown in Table 2. The PS states that "the irrigation using reclaimed wastewater should be stopped before three weeks of the yield time for the fruit crops and before two weeks for field and feeding crops" to take into consideration the sensitivity of some crops to the properties and the elements of treated wastewater. The PS permits the use of treated wastewater, without any restrictions on the irrigation activities, for harvesting the yields of Cotton, Luffa, Brooms and other crops that are not in direct contact with the public. However, concerning the crops that are in direct contact with the public through touch or eating, the PS imposes intensive preventive restrictions and protective procedures for using the treated wastewater in irrigation. The PS adopts eleven irrigation methods that enable the farming activities from using the reclaimed wastewater for irrigating specific types of crops. Each of these methods is classified as a two-way or a one-way restriction method (PS, 2003).

One-way restriction irrigation methods:

- A distance ≥ 25 cm above the ground between the dripping point and the crop or fruits.
- A distance ≥ 50 cm between the level of sprinklers and the fruits.
- A ground plastic cover between the treated wastewater and the fruits.
- Crops or fruits with a single crust or an uneaten shell.

Category	Reuse conditions	Exposed Group	Intestinal nematodes (arithmetic mean no. of eggs per liter)	Fecal coliforms (geometric mean no. per 100 ml)	Treatment to meet the micro-biological guidelines
А	Irrigation of crops probably to be consumed uncooked, sports fields, public parks	Farmers, consumers, public	≤1	≤ 1000	A series of stabilization ponds designed to attain micro- biological quality indicated, or equivalent treatment
В	Irrigation of cereal crops, industrial crops, fodder crops, pasture and trees	Farmers	≤1	No standard recommended	Retention in stabilization ponds for 8–10 days or equivalent helminths and fecal coliform elimination
С	Localized irrigation of crops in category B if exposure to workers and the public does not occur	None	Not Applicable	Not applicable	Pretreatment as essential by irrigation technology but not less than primary sedimentation

Table 1. WHO microbiological quality guidelines for wastewater use in agriculture (WHO, 2006).

Table 2. Classification of wastewater quality (PS, 2003).

Parameter	Unit	Class (A)	Class (B)	Class (C)	Class (D)
Quality	Туре	High	Good	Medium	Low
BOD <sub>5</sub>	mg/l	20	20	40	60
TSS	mg/l	30	30	50	90
Fecal coliform	CFU /100 ml	200	1,000	1,000	1,000
COD	mg/l	50	50	100	150
DO	mg/l	> 1	> 1	> 1	> 1
TDS	mg/l	1,200	1,500	1,500	1,500
рН	-	6.00 to 9.00	6.00 to 9.00	6.00 to 9.00	6.00 to 9.00
Fat, Oil & Grease	mg/l	5.00	5.00	5.00	5.00
Phenol	mg/l	0.002	0.002	0.002	0.002
Methylene Blue Active Substance	ma/1	15	15	15	25
(MBAS)	mg/1	15	15	15	25
NO <sub>3</sub> -N	mg/l	20	20	30	40
NH4-N	mg/l	5	5	10	15
Total-N	mg/l	30	30	45	60
Cl	mg/l	400	400	400	400
SO4	mg/l	300	300	300	300
Na	mg/l	200	200	200	200
Mg	mg/l	60	60	60	60
Ca	mg/l	300	300	300	300
SAR	-	5.83	5.83	5.83	5.83
PO4-P	mg/l	30	30	30	30
Al; Fe	mg/l	5	5	5	5
As; Cr	mg/l	0.1	0.1	0.1	0.1
Cu, Mn, Ni, Pb	mg/l	0.2	0.2	0.2	0.2
Se	mg/l	0.02	0.02	0.02	0.02
Cd	mg/l	0.01	0.01	0.01	0.01
Zn	mg/l	2	2	2	2
CN; Co	mg/l	0.05	0.05	0.05	0.05
Hg	mg/l	0.001	0.001	0.001	0.001
В	mg/l	0.7	0.7	0.7	0.7
E. coli	CFU /100 ml	100	1,000	1,000	1,000
Nematodes	Eggs/l	$\leq 1$	$\leq 1$	$\leq 1$	$\leq 1$

- Crops or fruits eaten only cooked.
- Sand filter detain the wastewater for at least 15 days or water ponds that contain less than 10% of treated wastewater.
- Disinfection by chlorine so that the residual chlorine is not less than 0.5 mg/l and the contact time is not less than an hour half or by using any other sterilization method.

Two-way restriction:

• A distance  $\geq$  50 cm above the ground between the dripping point and the crop or fruits.

Irrigation methods:

• Underground drip irrigation.

The PS requires specific standard considerations to irrigate crops by the reclaimed wastewater. The PS restricts the irrigation activities for gardens, playgrounds, and parks to be by a reclaimed wastewater quality of class (A). However, the processes of groundwater replenishment by infiltration and the offshore disposal of about 500 m into the sea should be by wastewater quality of not less than class (C). The standards permit the irrigation of crops of seeds production, dry feeds, forest trees, industrial corps



Figure 1 (a). Geographical location of the Gaza Strip, and (b) Positions of wastewater treatment plants in the Gaza Strip.

and grains using low quality of treated wastewater. The PS allow using treated wastewater with class quality of (A), or other quality with suitable one-way or two-way restriction irrigation methods to irrigate the crops of artichoke, eaten corns, citrus fruits, uneaten shell fruits, falling leaves trees, tropical crops, grapes, cactus, dates, olives, and flowers (PS, 2003). In the area of the Gaza Strip, four types of crops are cultivated and harvested: (a) field crops: wheat, potatoes, and onions; (b) vegetables: tomatoes, cucumbers, eggplants, squash, green beans, and paprika; (c) fruit trees: citrus fruits, guava, dates, and almonds; and (d) olives (ARIJ, 2015). Generally, drip irrigation remains the domain irrigation system and widely used for agricultural production by farmers in the Gaza Strip (ARIJ, 2015).

# 3. WATER ISSUES OF THE STUDY AREA

The Gaza Strip (shown in Fig. 1(a)) is a stretch of area that extends on the coast of the eastern Mediterranean with about 42 km long, between 6 and 12 km wide, and an area of 365 km<sup>2</sup>. The Gaza Strip is recognized as one of the most densely populated areas in the world. The estimations of the Palestinian Central Bureau of Statistics (PCBS) for the year 2018 revealed that the population of the Gaza Strip was more than two million inhabitants (PCBS, 2018). Concerning the current situation of the water sector, the region of the Gaza Strip is characterized by many parties as an area in a continuous humanitarian crisis due to the lack of both quantity and quality of water resources for different uses (PWA, 2011, 2013, 2014).

The underlying coastal aquifer is the only water resource that is exploited in the Gaza Strip for the domestic, industrial and agricultural uses. The status of the coastal aquifer shows permanent deterioration on both quantity and quality. The annual replenishment volume of the aquifer is between 55 and 60 million cubic meters while the

Palestinian abstraction from the aquifer in the Gaza Strip is quantified by about 180 million cubic meters per annum. The agricultural sector consumes the largest portion of nearly 50% of the entire pumped groundwater water from the aquifer comparing to the other remaining domestic and industrial sectors that tighter use the second half of the extracted groundwater. Accordingly, the records of water balance show a severe and continuous deficit of about more than 120 million cubic meters per annum (PWA, 2013). In terms of quality, the present rate of deterioration demonstrates that over 95% of the coastal aquifer is contaminated with unacceptably high levels of either nitrate (NO3) or chloride (Cl), posing significant health risks to Gaza's residents (PWA, 2014). The sustainability of coastal aquifer became a crucial and sensitive issue for the Palestinian Water Authority (PWA) and other Palestinian water-related organizations and agencies. Hence, in order to alleviate the huge pressing on the coastal aquifer, the PWA pays more attention to using new alternative and other nonconventional water sources.

i. Purchased water: the water sector in the Gaza Strip is supplied by 10 million cubic meters per year. This quantity is bought from Israeli water utility (Mekorot). In addition, under the negotiation's agreement of Oslo II, there is a commitment to increase the supplied quantity by 10 million cubic meters (PWA, 2014).

Desalination of seawater: desalination of seawater became a strategic option for the PWA to overcome the shortage in the water supply. Hence, the PWA provided a strategic plan for water supply along with the short and long terms. Nowadays, short-term low-volume (STLV) seawater desalination plants, the overall

capacity of 13 million cubic meters per annum, are built to enhance the quality and quantity supply of water in the Gaza Strip. From a long-term perspective, a plan to construct a regional strategic large-scale

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seawater desalination was adopted. The first phase of the regional seawater is intended to provide water sector by 55 million cubic meters yearly while the second phase will lift the plant's capacity to 120 million cubic meters per annum (PWA, 2015).

iii. Treated wastewater reuse: the reclamation of wastewater draws the attention of PWA as a potential source that could be used to maintain the coastal aquifer sustainable. Exploiting the reclaimed wastewater can achieve the sustainability of groundwater by 50%. Practically, the PWA starts studying the willingness of using reclaimed wastewater for irrigating agriculture (PWA, 2013).

On average, the water services suppliers estimate the total amount of produced wastewater in the Gaza Strip by approximately 50 million cubic meters per annum (ARIJ 2015; PCBS, 2018). Currently, there are four small scale and one large scale operating wastewater treatment plants, geographically demonstrated in Fig. 1(b), in the Gaza Strip. The four small scale wastewater treatment plants of Gaza, Wadi Gaza, Khanyunis and Rafah have the capacity to treat partially, and not in an acceptable level of quality, about 38.7 million cubic meters of the collected wastewaters in the Gaza Strip. This quantity which forms a ratio of about 75-80% of the total generated municipal effluents in the Gaza Strip is directly disposed into the marine ecosystem without enough proper treatment and without any mitigation measures. As well, the operating large scale of north Gaza (NGEST) wastewater treatment plant treats about 12.8 million cubic meters of wastewater which is utilized to recover the coastal aquifer through a number of wastewater infiltration basins.

The characteristic properties of raw wastewater quality for different governorates in the Gaza Strip, as depicted in Table 3, show that the effluent of wastewater has high organic matter, i.e. BOD<sub>5</sub> of 724 mg/l, compared with the average BOD<sub>5</sub> levels in the developing countries which range from 200-300 mg/l (Polprasert, 2007). These high pollutant contents affect adversely the treatment efficiency of the in-operation wastewater treatment plant. The high growth in the population of the Gaza Strip increases the load on the capacity and affects the initial quality design parameters that have been used to plan the current wastewater treatment plants which affect the treatment efficiency. Therefore, the sanitation services in the Gaza Strip are in curial crisis and the existing wastewater treatment plants work under overloaded flow rates and so some wastewater is being treated and the large amount is returned raw and pumped into the sea. Based on the design parameters of BOD, COD, and TSS, illustrated in Table 4, the designed efficiency of the small-scale wastewater treatment plants is about 75% (CMWU, 2012). However, nowadays, and due to over rates of wastewater and the continuous deterioration in the treatment efficiency of the small-scale wastewater treatment plants, the PWA suggested closing these plants and constructing instead three large scale wastewater treatment plants on the eastern border of the Gaza Strip (PWA, 2014). To enhance the status of public health and sanitation services, the PWA plans to replace the current over-loaded wastewater

treatment facilities by three new large-scale high-efficiency wastewater treatment plants located as shown in Fig. 1(b) in the North, Central, and South of the Gaza Strip. Recently, as shown in the provided timeline plan in Table 5, the wastewater treatment plant of North Gaza entered the service to replace the old plant at Beit Lahia. Also, the Gaza central and south wastewater treatment plants are under construction and the decision-makers confirm that they will be ready to operate by 2022. The efficiency of the three planned large-scale wastewater treatment plants will be very acceptable, where, the effluent characteristics are between 10 to 20 mg/l for BOD<sub>5</sub>, 15-20 mg/l for suspended solids, 10 to 15 mg/l for total nitrogen, less than 1 eggs per liter from Helminths and less than 200 CFU per 100 ml for Fecal Coliforms (PWA, 2011). The quality of effluent indicates that these strategic plants were designed to provide treated wastewater of class (A). According to the monitoring records of the PWA, the volume of treated wastewater that was used in 2012 is 1 million cubic meters which represent only 3% of the available partially treated wastewater. The PWA put a strategic plan, shown in Table 6, extends to the year 2032 to enhance the water management system in the Gaza Strip (PWA, 2013). According to this plan, by 2022, in the short-term strategy plan, it is expected to lift the exploited treated wastewater to more than 25% of the available partially treated wastewater. However, by 2032, the expectations of the long-term strategy plan address that about 25 million cubic meters per annum and nearly 75 million cubic meters of treated wastewater are going to be used for agriculture and aquifer recharging, respectively.

In order to alleviate the deterioration in the coastal aquifer, the long-term plan aims to decrease the overall groundwater abstraction in the Gaza Strip from the present rate of 180 million cubic meters per annum to about 70 million cubic meters per annum in 2032. As shown in Table 6, the long-term strategic plan aims to increase the sustainability of the coastal aquifer by using the reclaimed wastewater as a new water source for agricultural purposes. The plan expects that the total anticipated demands of water for agriculture will be reduced as a result of urbanization and population growth, hence, the projected potential irrigated lands will be about 90,000 dunums by 2032.

## 4. ATERIALS AND METHODS

This article highlights the performance of RO membrane technology as a post-treatment stage in enhancing the treatment efficiency to improve the quality of wastewater reclamation. The performance of this technology was investigated by setting up an experimental model and by testing and analyzing, in the laboratory, the quality of wastewater before and after the RO treatment. The experimental physical model, shown in Fig. 2, consists of: (1) Collecting tanks: High-density polyethylene (HDPE) feeder, permeate and concentrate tanks each of cubic meter volume were exploited to collect the raw and the reclaimed wastewater before and after the treatment process; (2) Sand filter: a composite of sand and well-graded sizes of gravels range from 2.37 and 9.5 mm was prepared to configure a cylindrical sand filter with a length of 50 cm and

Table 3. Characteristics of wastewater in the Gaza Strip (CMWU, 2012).

Parameter	Unit	Characteristics of wastewater according to area				
		North Area	Gaza	Rafah	Average	
Biochemical Oxygen Demand (BOD5)	(mg/L)	728	667	777	724	
Chemical Oxygen Demand (COD)	(mg/L)	1385	1306	1399	1,363	
Suspended Solids (SS)	(mg/L)	663	617	540	607	
SS/BOD	ratio	0.9	0.95	0.69	0.85	
BOD/COD	ratio	0.526	0.51	0.56	0.532	

Table 4. Efficiency of wastewater treatment in the Gaza Strip (CMWU, 2012).

Wastewater	Biochemica (BOD5)	al Oxygen	Demand	Chemical	Oxygen Den	nand (COD)	Suspende	d Solids (SS)	
Plant	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal
	mg/l	mg/l	%	mg/l	mg/l	%	mg/l	mg/l	%
Gaza	500	105	79	1020	220	78	550	110	80
Rafah	560	120	81	1160	255	78	550	122	79
KhanYunis	520	155	70	1090	322	70	580	141	76
Beit Lahia	440	133	70	980	250	74	480	222	71
Average	505	128	75	1,063	262	75	540	149	77

Table 5. Characteristics of large-scale wastewater treatment plant in Gaza Strip (PWA, 2011).

Facility	Flow (m³/day)	Current status	Future status	Final destination
North Gaza	35,000	North Gaza wastewater treatment plant; opened in 2018 to replace Bait Lahia treatment plant which is now out of service	Northern Gaza Emergency Sewage Treatment (NGEST) will be upgraded to a capacity of 70,000 m <sup>3</sup> /d	Infiltration Basins
Central	65,000	Gaza wastewater treatment plant; established in 1979; now the plant is overloaded with a capacity of 65,000 m <sup>3</sup> /day	Central Gaza treatment facility with a capacity of 65,000 m <sup>3</sup> /d to replace	Socuetor
Gaza	14,000	Middle Gaza wastewater treatment plant; established in 2014 with a capacity of 12,000 m <sup>3</sup> /day	the current plant; it will be operated in 2020	Seawater
South Gaza	14,000	Khan Yunis wastewater treatment plant; consists of three lagoons were built in to collect and treat wastewater during 2003 – 2009	South Gaza treatment plant with a capacity of 26,000 m <sup>3</sup> /d as phase I operated in 2019 and 44,000 m <sup>3</sup> /d operated in 2022 to replace the current treatment plant	Infiltration Basins

Table 6. Potential reuse of treated wastewater (1	PWA,	2013).
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Itoma	Years			
	2012	2022	2027	2032
Quantity of reclaimed wastewater	33.2	59.3	75.8	99.9
Irrigation portion (million m <sup>3</sup> /year)	1	14.8	19	25
Aquifer Recharge (million m <sup>3</sup> /year)	32.2	44.5	56.9	75
<b>Portion of groundwater for irrigation</b> (million m <sup>3</sup> /year)	86	59	45.5	32
Potion of constructed dams for Irrigation (million m <sup>3</sup> /year)	0	5	7.5	10
<b>Total Available quantity for Irrigation</b> (million m <sup>3</sup> /year)	87	78.8	72	67
Irrigation needs (million m <sup>3</sup> /year)	74.1	74.1	74.1	74.1
Irrigable land (dunum)	133,000	123,000	118,000	113,000
Potential irrigated land (dunum)	117,403	106,383	97,112	90,401
Percent of irrigable land (dunum)	88.3%	86.5%	82.3%	80%

a diameter of 30 cm to eliminate the content of large suspended solids before the RO unit; (3) Microfiltration (MF) and Ultrafiltration (UF) unit: are cellulose acetate semipermeable membranes that remove too large solids before the RO unit, the microfiltration membranes range from 0.1 to 10  $\mu$ m, and ultrafiltration membranes range from 0.1 to 0.01  $\mu$ m; and (4) RO unit: consists of a semipermeable membrane thin polyamide layer (< 200 nm) deposited on top of a polyethersulfone or polysulfide porous layer (about 50 microns) on top of a non-woven fabric support sheet. These semipermeable membranes have the ability to remove ions, molecules and larger particles from water by applying pressure to

overcome the osmotic pressure. Regarding, quality control and quality assurance (QC/QA), many experimental trials were executed during February and April of 2018 to take into consideration the seasonal change in the characteristics of wastewater that reaches the wastewater treatment plants in the Gaza Strip.

For each experimental trial, approximately 500 liters of wastewater sample was collected from the effluent of Gaza wastewater treatment plant and transported to the laboratory of the Islamic University of Gaza using barrels. The barrels were preserved into an icebox to inhibit the metabolic derangements of the wastewater organic content. In the laboratory, the collected wastewater samples from the Gaza

wastewater treatment plant were poured into the feeder tank. The wastewater was passed through the sand filter, which composes of graded-size gravel, the flow of wastewater inside the sand filter is governed by the force of gravity. The general design criteria for the slow sand filter recommend a filtration rate between 0.1 and 0.2 m per hour through the filter layers (Kawamura 2000). However, the filtration treatment through the designed sand filter demonstrated a slow sand filtration, hence, the water head reached about 20 cm above the sand filter layers with a filtration rate of 0.5 liters per minute. The passed wastewater through the sand filter is entered into the three stages of MF/UF membrane unit with 5- and 1-micron pores diameter which was selected as a second pretreatment unit after sand filter to protect the RO device from fouling and to reduce suspended solids. After sufficient pre-treatment, the water was pumped by a main pump of 130 psi (8.844 bar) through the RO unit with a flow of the filter was 1.8 liters per minute, the recovery rate was 50% from the fed water. In order to enhance the recovery efficiency of the system, a repass cycle was inserted into the system, where, a small repass pump of 4.23 bar was operated



Figure 2. Scheme of the experimental physical model.



Figure 3. Model configuration of Winflows.

to repump the concentrate through the RO unit. The operation of the repass cycle was designed to operate when the concentrate in the concentrate tank reaches a volume of 250 liters or more. Finally, the treated water i.e., permeate water, was collected into the permeate tank (5) which is the treatment product that was tested in the lab. For each experimental trial, four samples were taken in every stage of filtration in each of the two experimental trials for testing and analysis purposes. The samples were put in polythene bottles that were pre-washed with acid and distilled water and then were dried. The first sample was taken from the feeder tank and before sand filter, the second was taken after the sand filter, and finally, the last two samples were taken from RO concentrate and permeate tanks, respectively. The four gathered samples were preserved at 4°C in an icebox and brought to the testing department of the laboratory for testing and results declaration. The quality of the treated wastewater in every phase of the treatment process was tested in order to examine the efficiency of each component in the experimental model in terms of Biochemical Oxygen Demand (BOD), Total Suspended Solids (TSS), Total Dissolved Solids (TDS), Hydrogen Ion Concentration (pH), Electrical Conductivity (EC), Fecal Coliform (FC) and Nitrate (NO3-N). The described standard methods for the examination of water and wastewater by the American Public Health Association, American Water Works Association, and the Water Environmental Federation (2017) have been employed to examine the water quality parameters. The content of the organic matters has been tested in terms of the 5-day BOD test according to the procedure of the 5210 B. The described standard method in the procedure of 2540 D and 2520 B were applied to measure the TDS and the EC in the reclaimed wastewater samples, respectively. Furthermore, the acidity of the treated wastewater in term of pH was determined according to the procedure of 4500-H+. Concerning the pathogenic content, the FC concentration was detected according to the procedure of the 9211 B seven-hour fecal coliform test. Else, the concentration of NO3 in the wastewater was determined using the Nitrate Electrode method according to the procedure of 4500-NO3 D Practically, testing the efficiency of membrane technology as a post-treatment in wastewater reclamation from the view of quality is not sufficient to judge on the whole efficiency of this technology. Principally, the main challenge governs the development and the popularity of membrane technology is the challenge of energy. Thus, it is critical to estimate the feasibility of this system from the views of cost and energy consumption. It is difficult to address the cost and energy consumption through the experimental physical model. Hence, with the rapid increase in computer power, it is not surprising to note the highly dependent on numerical simulation as a prediction tool in the predesign and design stages. In this article, the simulation model of Win-flows RO system design and simulation software was used to estimate numerically the cost and energy consumption of wastewater reclamation through the designed experimental model. To ensure better simulation results, the software was run several times and a cartridge-filter-pre-treatment unit was added to the simulation system to provide a better

presentation of the reality. The Win-flows has some distinctive features like three pass systems, permeate split and recycle, anti-scalant dosing, energy recovery devices and ability to combine stages that make its ability higher than other similar software. The best presentation Windflows model for the experimental model of this study, shown in Fig. 3, was designed to treat 1,000 m<sup>3</sup> per hour of tertiary treated wastewater with a TDS of 3,800, pH of 7.8 at a temperature of 16°C. After a series of trials and errors, the final design of the model system consisted of Cartridge filter as pretreatment and two stages of RO elements, 90 pressure vessels, and 65 pressure vessels, every vessel contains 7 elements of Duraslick anti-fouling membranes that are designed especially for wastewater treatment. The selected membrane model was DSL RO8040 to maintain the continuity of membrane efficiency for 5 years. In the numerical model, the water is pumped through the system using two pumps. In the first treatment stage, the main pump, which has a pressure of about 18 bar draws the feedwater by a capacity of 505 m<sup>3</sup> per hour and pumps it into the RO membrane system. However, the rejected water from the first stage is mixed with a flow of feedwater and repumped again into the membrane system using a repass pump with a pressure of 4.23 bar and a capacity of 495 m<sup>3</sup> per hour. The numerical model demonstrates that the system gives a recovery ratio of 50% which is acceptable and similar to the recovery ratio of the experimental model.

## 5. RESULTS DISCUSSION AND COST ESTIMATION

Reusing of reclaimed wastewater is expected to overcome the deficit in the water management system as a new proper water source. The advance development in membrane technology supports the progress in reusing wastewater; however, the energy challenges could be the main obstacle to make these technologies practical. The applicability of RO membrane technology as a post-treatment unit to recycle wastewater was investigated based on the quality of the reclaimed wastewater and on the energy efficiency of the whole system. The quality of reclaimed wastewater was addressed by the experimental examination for the efficiency of using RO membrane technology in treating wastewater. The experimental trials and laboratory testing were performed several times to ensure better QC/QA and to present precise results and decisions. Table 7 illustrates the average removal efficiencies of impurities and pollutants through the RO experimental model in terms of different physical, chemical and biological parameters for all runs. The quality of collected wastewater from Gaza wastewater treatment shows higher concentrations than designed quality effluent by about 138%, and excess concentrations above the average designed concentrations for three operating wastewater treatment plant in the Gaza Strip by 95%. The tested samples provide practical evidence that the treatment plants in the Gaza Strip operate with an unacceptable quality level. Thus, this article demonstrates efficiency investigation for the applicability of using membrane technology to reclaim wastewater in a region suffers from inefficient wastewater treatment plants. The

depicted experimental model results demonstrate that the treated wastewater using RO membrane technology achieves acceptable water quality levels for the agricultural and groundwater replenishment activities. The quality of the treated effluent wastewater through the designed experimental model was examined in terms of the physical, i.e. BOD<sub>5</sub>, TSS and TDS, the biological, i.e. FC and NO<sub>3</sub>, and the acidity or basicity index of pH. In the light of BOD5, the whole system shows a high overall efficiency of 100% for organic contaminants rejection, and the efficiency of the sand filter and RO unit were 59% and 100%, respectively. The system units and the whole system reveal lower removal efficiency for TSS and TDS than other parameters. The capacity of the system in eliminating TSS was higher than that of TDS by about 5%. The sand filter contribution in the whole eliminating of TSS and TDS is insignificant and the efficiency of sand filter in removing TDS is almost 0%. The RO unit forms the main removing part by the efficiency of 97.5 and 92% for TSS and TDS rejection, respectively. The contents of FC in the tested wastewater samples were about within 1300 CFU per 100 ml. The ability of sand filters to eliminate the FC from the samples was about 95% and the RO unit shows full ability to remove the remaining FC after the primary treatment by a sand filter. The nitrification process takes a significant place in the experimental system; hence, the sand filter trends to increase the concentration of NO3 in reclaimed water as a result of conversion from NH4 into NO3 through the nitrification process. The NO3 is a rich nutrient to plant growth and it is considered as a valuable element to the agricultural activities. However, the RO unit has a high ability to remove NO3 and in all the whole system was able

to fully remove all NO<sub>3</sub> from the permeate.

Related to the pH, the nitrification process trends to covert the state of water into more alkalinity, hence, the results show that the pH of the treated wastewater rises from 7.7 to 8.7 after treatment so for the purposes of agricultural activities the treated wastewater must be adjusted to be within a pH value of 6.5 to 8.4 by adding a point entry injection system to pump an acid solution of acetic acid (white vinegar), citric acid or alum to reduce the basicity of treated water. The energy and cost estimations are two linked tasks, the specific energy consumption (SEC) for the RO system is commonly calculated using over simple analyses depending on the average operation tasks for the specific plant. The results of the built numerical model through the Winflows RO system design and simulation software indicate that the SEC of the whole experimental system is about 1.23 kWh per one cubic meter of permeate. However, the local membrane technology market indicates to set up a membrane-based treatment facility with a capacity of one million cubic meters of permeate per year over a five years lifespan is approximately 6 million United States dollars (USD) as shown in Table 8. Thus, the fixed cost estimation addresses that the loaded fixed costs on the unit volume of treated wastewater are about 0.4 USD per cubic meter of permeate. In the Gaza Strip, the cost of the municipal energy is approximated quantified by 0.15 USD for 1 kWh. Hence, the total cost to reclaim one cubic meter of wastewater is about 0.6 USD. Reviewing the cost and energy consumption estimations for similar RO wastewater treatment plants worldwide could introduce better indication about the expected cost and energy to produce one cubic meter of reclaimed wastewater. Facing all the challenges

Table 7. 1	Experimental	results of RO	wastewater reclamation.
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	BOD <sub>5</sub> (mg/l)	TSS (mg/l)	TDS (mg/l)	pН	FC (CFU /100 ml)	NO <sub>3</sub> (mg/l)
Raw wastewater	250	1,070	3,800	7.7	1,250	0.4
After sand filter	103	837	3,800	7.8	67	15
Concentrate	230	1,700	7,300	7.8	10	2
Permeate	Nil	20	296	8.7	Nil	Nil
Sand filter efficiency (%)	59	22	0	Increases pH	95	Increases NO <sub>3</sub>
RO-unit efficiency (%)	100	97.5	92	Increases pH	100	100
System efficiency (%)	100	98	92	Increases pH	100	100

Series	Item	Unit	No.	Unit Price (USD)	Total price (USD)
1	8-inch low fouling DUASLICK membrane	No.	1,085	1,900	2,061,500
2	8-inch vessels (7 elements per vessel)	No.	155	4,000	620,000
3	Cartridge filter	No.	8	3,000	24,000
4	Primary pump	No.	10	5,000	50,000
5	High pressure pump	No.	10	15,000	150,000
6	Pressure exchange	No.	2	50,000	100,000
7	Dossing pump with tanks	No.	2	10,000	20,000
8	Pressure exchange	No.	2	50,000	100,000
9	Dual media filters	No.	4	7,000	28,000
10	Backwash pumps	No.	4	3,000	12,000
11	Flow meters	No.	4	1,000	4,000
12	Skids	No.	1	30,000	30,000
13	PLC	No.	1	200,000	200,000
14	Fittings and connections	No.	1	100,000	100,000
15	$H_2SO_{4 along}$ five years	kg.	45,000	2	90,000
16	Hanger and other plant structure	No.	1	750,000	750,000
17	Effluent and permeate tanks 4000 m <sup>3</sup>	No.	2	800,000	1,600,000
18	Total price	USD	-	-	5,939,500
19	Amount of permeate along five years	m <sup>3</sup>	-	-	15,000,000
20	Total cost per 1 m <sup>3</sup> of permeate	USD			0.394

and constrains relating to using RO in the reclamation of wastewater as new technology, the main two largest membrane-based treatment plants in the world, Sulaibiya wastewater treatment plant in Kuwait and Orange Country wastewater treatment plants in the United States (USA) produce daily hundreds of thousands cubic meters of reclaimed wastewater with stability and continuance. Sulaibiya wastewater treatment plant was opened in 2004; the plant is the biggest membrane-based water treatment plant. Currently, the capacity of SULAIBIYA wastewater treatment plant is about 375,000 m<sup>3</sup> per day and it is planned to expand it to reach a capacity of 600,000 m<sup>3</sup> per day and it is anticipated to cover 26% of Kuwait's gross water needs, and is expected to reduce the demand from 140 to 25 million cubic meters per annum (Pearce, 2008). The Orange Country plant was opened in 2004, the facility treats 320,000 m<sup>3</sup> daily of wastewater effluent using RO membrane technology. The plant is going to be expanded to a capacity of 590,000 m<sup>3</sup> per day (Water Technology, 2018). Generally, the estimations reveal that the conventional treatment of wastewater by the RO membrane technology, the costs about 85 to 90 cents per cubic meter of permeate (Pearce, 2008; Water technology, 2018). On the other hand, the requirement of energy to purify water depends on the quality of raw water and the applied treatment technique. The energy consumption for the purification of natural sources such as surface water and groundwater using membrane filtration is about 0.1 kWh per cubic meter. However, the energy consumption for treating surface water using both conventional treatment and UF/MF membrane is estimated by about 0.25 to 0.35 kWh per cubic meter. In general, the energy of water treatment could be estimated based on the TDS of the raw water, for TDS of less than 3,000 ppm about kWh of energy is needed to purify one cubic meter however for TDS between 3,000 and 11,000 ppm nearly 1.7 kWh of power is required to treat a cubic meter of water. Regarding, wastewater reclamation the energy requirement to recycle wastewater using dual membrane filtration of MF/UF and RO needs from 0.8 to 1.2 kWh per cubic meter. However, the use of a membrane bioreactor followed by RO requires energy between 1.2 and 1.5 kWh per cubic meter. In the countries of the Mediterranean Sea, the RO seawater desalination technique consumes about 2.3 to 4 kWh per cubic meter of freshwater (Pearce, 2008).

## 6. CONCLUSION

Reclamation of wastewater is a promising source to meet the increasing water demands in the arid and semi-arid regions. The agricultural sectors consume more than 50% of the water budget and providing treated wastewater with acceptable quality levels for irrigation can increase the sustainability of the water system and the fertility of agricultural lands. The rapid advance in the membrane technology draws the attention toward using RO membrane technology as a post-treatment process for wastewater treatment. The pollutants removal efficiency of RO technology, as well as the low cost of operation and maintenance, makes this technology dominant in water treatment. This study experimentally concludes that the ROmembrane technology system has the ability to remove more than 90% of solids pollutants and the efficiency of eliminating the other bacterial and ionic pollutants could reach 100%. The study estimates the energy consumption for wastewater treatment using RO by around 1.23 kWh per cubic meter of permeate which is far less than the consumption of other alternative solutions brackish water and seawater desalination that consume about 1.7 kWh and up to 4 kWh per cubic meter, respectively. This technology provides a sustainable treatment process for countries suffering from a lack of electrical sources since the experimental model of this study estimates that the fixed cost to treat one cubic meter of permeate costs approximately 40 cents, compared to other comparison studies, the described model of this study could save more than 50% of the fixed costs to treat one cubic of wastewater.

Therefore, the findings of this study clarify that the ROmembrane technology system as a post-treatment stage in the wastewater treatment plants of the Gaza Strip could improve the wastewater quality to meet PS guidelines and produce a reclaimed wastewater of class (A) that could be used without restrictions to irrigate gardens, playgrounds, parks, groundwater replenishment and for the offshore disposal at about 500 m into the sea. Moreover, the quality of the reclaimed wastewater could be used for planting crops of seeds production, dry feeds, forest trees, industrial crops and grains, crops of artichoke, eaten corns, citrus fruits, uneaten shell fruits, falling leaves trees, tropical crops, grapes, cactus, dates, olives, and flowers. Regarding the existing drip irrigation system in the Gaza Strip which is widely common, this study recommends the use of the reclaimed wastewater using RO membrane technology to irrigate the crops of citrus, guava, dates, almonds, and olives under a two-way restriction measures by lifting the irrigation system by at least 0.5 meters from the ground. In conclusion, the applicability of the RO treatment system could be a significant component to enhance the strategic water development plan of the PWA to maintain the sustainability of the coastal aquifer.

## **CONFLICT OF INTEREST**

The authors declare no conflicts of interest.

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## IDENTIFICATION OF OBSTRUCTIVE SLEEP APNEA USING ARTIFICIAL NEURAL NETWORKS AND WAVELET PACKET DECOMPOSITION OF THE HRV SIGNAL

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Abstract: The advancement of telecommunication technologies has provided us with new promising alternatives for remote diagnosis and possible treatment suggestions for patients of diverse health disorders, among which is the ability to identify Obstructive Sleep Apnea (OSA) syndrome by means of Electrocardiograph (ECG) signal analysis. In this paper, the standard spectral bands' powers and statistical interval-based parameters of the Heart Rate Variability (HRV) signal were considered as a form of features for classifying the Sultan Qaboos University Hospital (SQUH) database for OSA syndrome into 4 different levels. Wavelet packet analysis was applied to obtain and estimate the standard frequency bands of the HRV signal. Further, the single perceptron neural network, the feedforward with back-propagation neural network and the probabilistic neural network have been implemented in the classification task. The classification between normal subjects versus severe OSA patients achieved 95% accuracy with the probabilistic neural network. While the classification between normal subjects versus mild OSA subjects reached accuracy of 95% also. When grouping mild, moderate and severe OSA subjects in one group compared to normal subjects as a second group, the classification with the feedforward network achieved an accuracy of 87.5%. Finally, when classifying subjects directly into one of the four classes (normal or mild or moderate or severe), a 77.5% accuracy was achieved with the feedforward network.

Keywords: Sleep Apnea; Identification; Classification; HRV; Wavelet Packet Decomposition; Neural Networks.

## تحديد انقطاع التنفس الانسدادي أثناء النوم باستخدام الشبكات العصبية الاصطناعية وتحليل حزمة الأطوال الموجية لإشارة HRV

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**الملخص:** أتاح لنا التقدم العلمي في مجال تقنيات الاتصال بدائلا جديدة واعدة للتشخيص عن بعد واقتراحات علاجية محتملة لمرضى مختلف الاضطرابات الصحية. ومن ضمن هذه التقنيات القدرة على كشف مرضى متلازمة انقطاع التنفس الانسدادي أثناء النوم عن طريق تحليل إشارة التخطيط الكهربائي للقلب (ECG). اعتبرت في هذه الدراسة قوى النطاقات الطيفية القياسية والإحصائيات المقسمة على فترات زمنية لإشارة تغير معدل نبضات القلب (HRV) سمة من سمات مصنيف وتقسيم قواعد بيانات مستشفى جامعة السلطان قابوس المتعلقة بمتلازمة انقطاع التنفس (HRV) محتملة من سمات محسوباتي لقلب (ECG). اعتبرت في هذه الدراسة قوى النطاقات تصنيف وتقسيم قواعد بيانات مستشفى جامعة السلطان قابوس المتعلقة بمتلازمة انقطاع التنفس (HRV) سمة من سمات محسوبات. تم إستخدام حزمة الأطوال الموجية لتقدير نطاقات التردد القياسية لإشارة تغير معدل نبضات القلب (HRV) سمة من سمات محسوبات. تم إستخدام حزمة الأطوال الموجية لتقدير نطاقات التردد القياسية لإشارة والاتشار الخلفي، والشبكة العصبية الحسية الحيية الحصية الحميية وتقسيم كل من الشبكة العصبية الوحيدة، و الشبكة العصبية ذات التغذية الأمامية والانتشار الخلفي، والشبكة العصبية المرضى من ذوي الأعراض وتصنية بعن معميم كل من الشبكة العصبية الحسية الوحيدة، و الشبكة العصبية بن الأصحاء والمرضى من ذوي الأعراض الحصبية الإحتمالية, ودقة 95% في التمييز بين الأصحاء والمرضى من ذوي الأعراض الحادة باستخدام نموذج الشبكة العصبية الإحتمالية, ودقة 95% في التفريز بين الأصحاء والمرضى من ذوي الأعراض الحادة باستخدام نموذج الشبكة العصبية الإحتمالية, ودقة 95% في التفريز بين الأشحاص الأصحاء والمرضى من ذوي الأعراض الحادة باستخدام نموذج الشبكة العصبية الحتمالية, ودقة 75% في التفريز بين الأشحاص الأصحاء والأسخاص المصابين الحادة باستخدام نموذج الشبكة العصبية الإحتمالية, ودقة 75% في التفريز بين الأسحاء والمرضى مناز من أعراض من أعراض معنون من أعراض بسيطة. أما عند تشكيل عينة تشمل مرضى يعانون من أعراض مخلفي المحابي والموبي والأسرة ويعانون من أعراض بسيطة. أما عند تشكيل عينة تشمل مرضى يعانون من أعراض مخلفي والإسخان معميه والمربي والغ مراض مخلية والمرغرية والموبن والغ مالمرية والغربي مرمال مرضى مرموذ الشبكة العصبية ذات التغذية الأمامية والإنفي والأممان ومولي والموبي والغي مانمرذا مالغفي مانمان

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#### **1. INTRODUCTION**

A sleep disorder occurs when the pattern of sleep is interrupted repeatedly during sleep (Kumar V 2008). Lack of sleep results in abnormalities in functions of the brain leading to cognitive impairment, changes in mood, low productivity, daytime sleepiness and abnormal hormonal rhythms (Wilson S 2016). Sleep apnea is a chronic disease that affects the health and productivity of individuals (National Heart, Lung and Blood Institute 2016) since it causes abnormal sleep pattern. Obstructive Sleep Apnea (OSA) is the most common type of sleep apnea followed by Central Sleep Apnea (CSA) and Mixed Sleep Apnea (MSA). OSA affects 3~4% and 2% of middle-aged men and women respectively (Lee W et al. 2008). Unlike CSA which results from heart failure or brain disorders, where the brain fails to control breathing leading to cessation of all respiratory airflow and movements, OSA results from a repeated process of complete or partial collapse in the upper airways of the respiratory system ranging from few seconds (minimum 10 sec) to minutes despite the ongoing brain efforts for the body to breath. The OSA events may occur more than 30 times and up to 100 times per hour. MSA on the other hand, is a mixture of CSA and OSA in the same individual. (American Academy of Sleep Medicine 2001; National Sleep Foundation 2016). OSA has been related to some serious co-morbidity such as cardiovascular diseases, arrhythmia, strokes, obesity, depression, certain types of hypertension and type 2 diabetes mellitus (Global Leaders in Sleep and Respiratory Medicine 2013; Xie W et al. 2014). There are several screening methods used for OSA detection to find evidence of its presence in patients for further evaluation. These methods depend on psychometric and physical evaluations during the routine health check-ups. Polysomnography (PSG) sleep study is the gold standard test for sleep apnea diagnosis. This test requires the patient to sleep in a sleep laboratory while attached using several electrodes to many devices for different biometric measures carried out by qualified sleep physicians overnight. The severity of sleep apnea is commonly determined by an Apnea-Hypopnea Index (AHI) which represents the number of obstructive, central, mixed and hypopnea episodes occurring during an hour of sleep (American Academy of Sleep Medicine 2001). If the AHI ranges between 0-5 apneic episodes during an hour of study time or sleep then the condition is considered normal. An index of 5-15 is considered mild while an index of 15 - 30 is considered moderate and if the index is 30 or above, the subject is considered to have a severe degree of sleep apnea (Global Leaders in Sleep and Respiratory Medicine 2013). Electrocardiography (ECG) is a method used to measures the electrical activity of the heart by placing electrodes on different parts of the body (WebMD 2016). A normal sinus rhythm reflects the normal activity of the heart while

pumping blood to perform the sympathetic and parasympathetic activities (UCDavis Health System 2016). A typical ECG signal is produced when the heart chambers contract and expand to pump oxygenated-blood throughout the body and circulate the desaturated blood to the lungs. Fig. 1 (a) shows a typical ECG signal (Sharma S. *et al.* 2019).

Heart rate (HR) is a simple measurement that indicates the average number of heart beats during a certain time period (usually, a minute). A low HR reflects resting status while high HR indicates stress or exertion (Moore J 2016). Heart Rate Variability (HRV) on the other hand is a measure of the time variability in milliseconds between consecutive beats or correspondingly in the instantaneous HR. In other words, variation analysis of instantaneous HR versus time axis. HRV is sometimes called the R-R interval (RRI) analysis, where R is the peak point of the QRS complex in the ECG wave, or the Inter-Beat-Interval (IBI) analysis. When the individual is at rest, high HRV is favorable while low HRV is observed at an active or stressed state. HRV has been used as a measurement to assess overall cardiac health and reflect the state of the Autonomic Nervous System (ANS) activities (Hamilton G. et al. 2019).

The ANS is the involuntary division of the nervous system and consists of autonomic neurons that conduct impulses from the central nervous system (brain and/or spinal cord) to glands, smooth muscles and cardiac muscles (DanTest Clinicians Team 2016). The role of the ANS is to continuously fine-tune the functions of organs and organs systems to maintain internal stability and balance. ANS has two main components called the Sympathetic and Parasympathetic Nervous Systems (SNS and PSNS respectively). The SNS triggers the fight or flight response leading to increased heart rate, blood pressure and sweating, and pupil dilation etc. On the other hand, the PNS complements the operations



**figure 1: (a)** Normal ECG signal. **(b)** ECG signal at Apnea Episode.

performed by the SNS and triggers the rest and digest response where the opposite behavior occurs. When the airway is partially or completely obstructed; the heart rate changes and hence ECG signal alters. When the oxygen level decreases in the body during sleep apnea event the heart cells receives less oxygen and hence the heart rate is reduced and the R-R interval increases between consecutive beats as shown in Fig.1 (b) (Sharma S. *et al.* 2019).

This alters the brain's sleep and wakes the brain for immediate action. The brain responds by sending strong tones to the respiratory system to increase breathing speed. The later increases the heart rate suddenly and hence increases the blood pressure in order to pump more blood to compensate for the lack of oxygen.

In frequency-domain analysis, signals can be represented in a graph that shows how much (energy) of the signal lies within given frequency bands over a range of frequencies. The well-known Fourier methods such as the Fast Fourier Transform (FFT) implementation of the Discrete Fourier Transform (DFT) are usually used for identifying the available spectral content for both stationary and non-stationary signals (Polikar R 2011). However, for non-stationary signals, the frequency content varies with time and hence DFT based methods fail to provide the timerelated information at which those frequencies occur. Moreover, it can only reflect the frequencies that are present in the signal but not when they were present. Since most of the physiological signals like the ECG, etc. are non-stationary signals; time-frequency analysis such as the Short-Time Fourier Transform (STFT) and Discrete Wavelet Transform (DWT) are used as alternatives to the Fourier analysis when estimating the available PSD content (Polikar R 2011). The classification features used in this research depend on the Power Spectral Density (PSD) at different frequency levels estimated by implementing the Discrete Wavelet Packet Decomposition (DWPD) method. This is to overcome the resolution related problems of the STFT. The discrete wavelet decomposition utilizes various mother wavelets of different scales to be able to adapt to fast and slow changes in the analyzed signal (Polikar R 2011). The wavelet decomposition method is implemented using filter banks (Misiti M et al. 1996). A set of high pass and low pass filters allow the signal to be decomposed reaching a certain decomposition level in which the signal can be further analyzed, de-noised or compressed (Misiti M et al. 1996). The PSD calculation is done by mathematical modulation to the filters output coefficients (Sysel P et al. 2008). The DWT allows only the low-frequency components of the low pass filter to be analyzed to further levels as they are thought to be the ones that carry important information (Misiti M et al. 1996). However, DWPD allows both outcomes of the filters (low and highfrequency) to be further decomposed. The later emphasizes the outliers, edges and transient signals

which are crucial to tackle the OSA episodes. Hence, the DWPD leads to finding more desirable features for classification applications.

In time domain analysis, a signal instance's real values are visualized. A time domain graph shows how a signal changes with time. OSA episodes can be analyzed by observing the cyclic length variability of the HRV (a.k.a. RRI) signal. The term NN is used sometimes in place of the RR to emphasize that normal beats are being processed. There exist multiple well-established features that are normally used to analyze the beat-to-beat intervals (Mietus J et al. 2002). These features include: the Root Mean Square of Successive Differences (RMSSD), the Standard Deviation of Successive Differences (SDSD), the Standard Deviation (Std.) of entire RRI signal, the mean of the entire RRI signal, the NNx family measures which include (NN of  $x \le 50$ : NN50, NN30, NN20...etc.), and finally the pNNx family measure.

## 2. ECG DATABASE

It has been suggested by some researchers, that the uniqueness of the data sets affects the classification results of the different proposed methods, hence similar results cannot be obtained using different datasets (Lado M et al. 2011). In this research the ECG signals were collected from the Sleep Laboratory of the Physiology Department of the Sultan Qaboos University Hospital (SQUH) while performing PSG studies for 80 subjects. These records were obtained from 20 normal subjects (0<AHI<5), 20 mild subjects (5<AHI<15), 20 moderate subjects (15<AHI<30) and 20 severe subjects (AHI≥30). The records were divided into two groups: a training set and a test set; where both of the sets comprise of 10 normal ECG signals and 10 Apneic ECG signals from each of the mentioned groups (total of 40 signals per set). Features were extracted from both sets and the training set is used to train the neural networks for classification purpose while the test set was used to check the classification performance.

## 3. RRI EXTRACTION

The HRV signal is generated by finding the R-to-R Intervals (RRI) from the original ECG signals as declared in Fig. 1 (a). The proposed method in (Al Ghunaimi B 2003) was used to generate the RRI data. In order to accurately identify those R peaks, QRS detection is to be carried. The QRS detector that was used in (Al Ghunaimi B 2003) is a part of the Physionet tools available in the Physionet website and is based on the Pan-Tompkins Algorithm. Intervals corresponding to Normal-to-Normal peaks are extracted. The generated RRI data could contain false intervals, missed intervals and/or ectopic intervals. False RR intervals were removed by setting the lower and upper limits of its values to ones found in normal subjects which are typically around 400-2000 milliseconds. Removing of outliers was achieved using a 41-points Moving Average Filter (MAF). Resampling at 1 Hz and substituting of missed peaks were then achieved by simple linear interpolation implemented by MATLAB (Al Ghunaimi B 2003). Re-sampling and estimation of missed value are intended to generate an equally spaced RRI data and preserve the temporal sequence that is necessary for the frequency domain analysis. At this point it can be assumed that a clean RRI data sampled at 1 Hz containing no missed or outlier values was generated.

## 4. SPECTRAL FEATURES

In this research, the RRI signals were decomposed using discrete wavelet packet decomposition up to 9 levels in order to define the VLF band precisely according to the standard definition (VLF starts at 0.0033 Hz). At the ninth level, the signal would have been decomposed into 512 frequency bands including the low-frequency and high-frequency bands.

The PSD summation of these bands provides the total power spectrum of the signal, while the PSD summation by grouping according to certain frequency bands would provide us the desired features for the analysis. The discrete RRI signals are sampled at Fs=1 Hz and the maximum spectral frequency of RRI is found by Fs/2=0.5 Hz. Using DWPD, the 0.5 Hz is decomposed into 512 bands (9 levels =  $2^9$ ), while each band covers 0.5/512= 0.0009765625 Hz. Therefore, the standard spectral bands of the decomposed RRI are covered as shown in Fig. 2. In addition, the spectral values near zero Hz have the most energy content that dominates other spectral values. Therefore, we intend to exclude those spectral values below 0.0033 Hz; hence, the first three bands of the decomposed RRI signal were ignored and the PSD of the VLF was estimated starting from the 4<sup>th</sup>band at 0.00390625 Hz up to 0.040039063 Hz.

Fig. 3 shows the spectral powers summation of the different bands defining each feature. Furthermore, three other features extracted from the power ratios of the VLF, LF and HF features are calculated to form a total of six spectral features. These ratios are described as in Fig. 4.

In Fig. 5, the power of the HF band is sketched for both normal and severe subjects for the training set. The values of the HF band at normal subjects are higher than those of severe subjects. However, some severe subjects have powers that overlap with normal subjects resulting in difficulties to classify and hence a combination of features are to be investigated (The University of Nottingham 2017).

## 5. STATISTICAL FEATURES

In this work, multiple features were calculated from the RRI signals in time domain using MATLAB software. These features include the Root Mean Square of Successive Differences (RMSSD) which is calculated by finding the square root of the mean of the successive differences between adjacent NNs. The Standard Deviation of Successive Differences (SDSD) where the Standard Deviation of Average NN Intervals (SDANN) is calculated over a short period of 5-minutes or so to measures the change in heart rate due to cycles longer than five minutes. The Standard Deviation (Std.) of entire RRI signal, the mean of the entire RRI signal, The NNx family measures which include (NN of  $x \le 50$ : NN50, NN30, NN20 etc.), where it represents the number of pairs of successive NN's that differ by more than x=50, 30,20...etc. milliseconds and finally the pNNx family measure which includes the NNx measure divided by the total number of NNs of the signal. To sum up, the features are: the RMSSD, pNN15, pNN20, pNN30, pNN50, NN15, NN20, NN30, NN50, SDANN and Standard deviation of the entire signal which equate to eleven features.

In order to observe a relation between time domain and frequency domain features, the values of RMSSD, NN50, and pNN50 features were normalized by dividing them to the total power of the RRI signal as shown in Fig. 6. Similar behavior of Fig. 5 was observed where the power of the highfrequency band for severe apnea level subjects decrease and the same features' values increase for normal subjects. This allows us to use these features in a similar analogy as spectral features for representing the parasympathetic activity of the Autonomous Nervous System for instance. Of course, variations are also present in some of the OSA patients where their spectral or time domain features exhibit similar behavior as normal subjects making it more difficult to differentiate between the cases using a single feature.

## 6. NEURAL NETWORKS

The Artificial Neural Networks (ANNs) were designed based on the rudimentary understanding of the biological nervous system back in the 1950s to help solving and computing any arithmetic or logical statement (Hagan M *et al.* 1996). The ANNs are a collection of computational units called neurons composed of inputs with weights, biases and transfer functions to perform the thresholding act and produce an output. The three networks used in this work are of supervised learning type; this is because we already have information about the subject's original condition (normal, mild, moderate, and severe). In supervised learning, the training set consists of



Figure 2. Frequency bands of RRI packet decomposition into 9 levels.

$$VLF Power = \sum_{n=1}^{41} PSD(n)$$

$$VLF Power = \sum_{n=1}^{41} PSD(n)$$

$$VLF Power = \sum_{n=42}^{154} PSD(n)$$

$$(1)$$

$$LF Power = \sum_{n=42}^{154} PSD(n)$$

$$HF Power = \sum_{n=42}^{410} PSD(n)$$

$$(2)$$





Figure 4. Main three spectral ratio features.





n=155

(3)

Figure 5. Power spectral destiny (HF band) between normal and sever subjects.



multiple training examples each is a pair of input (feature) and the desired output (target). The supervised learning algorithm then tries to process and analyze the training examples to produce an inferred function (classification boundary region), which can be used to map new examples. If the network training and the learning algorithm are performed well, an optimal scenario would be generated in which the network will generalize and be able to determine the class label (target) of unseen instances correctly (Mohri M *et al.* 2012). This section introduces the three artificial neural networks' models used in this research and describes their learning algorithms and transfer functions briefly.

The perceptron network refers to multiple-inputs single-neuron network. It is considered the building block of a more complicated network called the Feed forward Neural Network. Usually the single-laver perceptron is used for binary and linear classification. The feed forward network is one of the most commonly used artificial neural networks and is typically composed of multiple perceptrons aligned in layers. It is considered the first type of ANN's that was used to solve non-linear problems where the perceptron has failed and is considered powerful networks that can almost approximate any function (Hagan M et al. 1996). Probabilistic Neural Networks (PNN's) are widely used classification and pattern recognition. When employed for classification problems, the class probability of input is estimated and the class with the highest probability is selected as the output class. This means that the input belongs to the class that provides the highest probability when introduced to that input. The design of a PNN is straightforward and extremely less complicated than other multilayer networks. This is because it does not depend on weights learning and hence does not require training. A PNN network generalizes well and is guaranteed to converge to a Bayesian classifier (simple well-studied conditional probabilistic classifier) providing the correct probability when presented with enough training data samples. A PNN consists of several sub-networks, each of which is a Parzen window pdf estimator for each of the classes. The inputs are the set of measurements/features and are used as centers for the radial basis (Gaussian functions) of the second layer. The third layer performs an average operation of the outputs from the second layer for each class and a final voting is performed by the third layer selecting the largest value to determine the associated class label.

In this research, the perceptron network is composed of one neuron of multiple inputs for one output classification, and two neurons for four outputs classification embedded with hard limit transfer function. The feedforward network is composed of two hidden layers each of five neurons (total 10) of the tangent-sigmoid transfer function and an output layer of one or two neurons according to the number of outputs (similar to perceptron layer) with pure linear transfer function. The probabilistic network is composed of input neurons changing according to the number of inputs with radial basis transfer function. Figs. 7 and 8 show the connections of the layers for the feed forward and probabilistic neural networks.

The performance of the training step and testing step of the neural networks are investigated by calculating well-known performance metrics such as the specificity, sensitivity and accuracy. The specificity reflects the number of accurately diagnosed healthy subjects while the sensitivity reflects the number of the accurately identified patients. The accuracy is a measure of both of the correctly classified patients and normal among the total experiment set. In Fig. 9, the actual meaning for each performance metric can be observed while the following equations reveal how they are calculated.

Specificity (%) = 
$$\frac{TN}{TN + FP}$$
.100 (1)

Sensitivity (%) = 
$$\frac{TP}{TP + FN}$$
.100 (2)

Accuracy (%) = 
$$\frac{TP + TN}{TP + FP + TN + FN}$$
.100 (3)



Figure 7. Feedforward neural network layer connections.



Figure 8. Probabilistic neural network layer connections.



Figure 9. Networks performance metrics.

### 7. IMPLEMENTATION

In this work, four different versions of classification have been carried out using the perceptron network, the feedforward with back-propagation network and the probabilistic neural network. These classification versions include:

- Version 1: Normal vs. Severe Classification
- Version 2: Normal vs. Mild Classification

• Version 3: Normal vs. Patient (Mild, Moderate, Severe) Classification

• Version 4: Normal vs. Mild vs. Moderate vs. Severe Classification.

The discrete wavelet packet decomposition was implemented to extract power estimations of the RRI signals of SQUH ECG database at the classical frequency bands with Bi-orthogonal mother wavelet at 9 levels decomposition. Four different schemes of training and test data are selected:

- Scheme 1: Original first half data for trial: Original second half data for the test.
- Scheme 2: Original second half data for trial: Original first half data for the test.
- Scheme 3: Large set simulated from first-half data for trial: Original second half data for test.
- Scheme 4: Large set simulated from second-half data for trial: Original first half data for test.

The networks in scheme 1 and 2 are trained by the features of original data sets containing 10 subjects for each of the normal, mild, moderate and severe OSA states. While in scheme 3 and 4, the networks are trained using a large simulated set generated from the original data sets. The large training set was acquired by generating randomly, 1000 uniformly distributed feature sets between the maximum and minimum values of each feature from the original extracted spectral features. The networks testing and performance computation were carried by the remaining equivalent-size original data set of 10 subjects in each case.

The statistical time-domain features are used with classification version number 4 only as it is the most complicated classification version and hence investigation with different features may increase the chance of enhancing the performance.

## 8. RESULTS AND DISCUSSION

Figures 10 and 11 display the actual classification accuracies of every network performing version-1 classification of normal vs. severe OSA conditions and version-3 normal vs. patient OSA conditions respectively. The spectral features used are numbered in Figs. 11 and 12 as: (1. VLF, 2. LF, 3. HF, 4. VLF/LF, 5. VLF/HF, 6. LF/HF). The patient-class refers to a set including all the apneic levels of mild, moderate and severe subjects where the AHI index is exceeding 6 and up to 100 or more. The features were selected based on the highest training results they provided before testing the networks as well as for their lowest discrepancy among other schemes.

Figures 12 and 13 summarize the results of the networks at versions-2 and version-4 classification by emphasizing the spectral features generating the highest performances. It can be noticed that version-4 classification was the most difficult and the results are extremely poor. Herein, it can be concluded that spectral features alone are not enough for this complicated task. However, when using statistical time-domain features with the feedforward network; the result is extremely improved reaching an accuracy of 77.5% using two features only. The features are of pNN20 & pNN30 respectively. This shows that the statistical features extracted from the RRI signal at the time domain used with the feedforward network were able to generate a non-linear decision surface that helped classifying those subjects unlike the probability estimates or linear classification boundaries generated by the probabilistic and perceptron networks respectively. It can be observed that the feedforward with back-propagation neural network achieved high accuracies at different classification versions demonstrating the power and consistency of the network. Further, the specificity and sensitivity percentages are very close to each other at the different classification versions of the different networks which implies that only few unique cases were miss classified. Table 1, summarizes the results of the networks at the different classification versions by emphasizing the features generating the highest performances using scheme 1 of training. The classification performance between normal individuals and severe OSA patients was highest using the probabilistic network where the network was able to classify the subjects into either normal or severe conditions at 95% accuracy. This is an interesting result and shows how powerful the probabilistic network can be even when trained with a few sample points; it was still able to generalize well. Moreover, this accuracy was achieved when using a single spectral feature which is the very low frequency power feature. The test set included 20 subjects (10 normal and 10 severe) meaning that the network only missed the correct classification of one severe subject at a sensitivity of 90%.



Figure 10. Networks Classification Results for Normal vs. Severe.



Figure 11. Networks classification results for normal vs. patient.



Figure 12. Networks classification results for normal vs. mild.



Figure 13. Networks classification results for normal vs. mild vs. moderate vs. severe.



Figure 14. Neural Networks Topology for the Four Classification Versions.

The second classification version between normal and mild OSA patients witnessed its best performance at 95% accuracy by the feedforward network among all training schemes (original and large training sets). Hence, it can be considered a robust network algorithm, since many examples were introduced to the network that included different types of apneic episodes exhibiting different cessation occurrences and durations. Moreover, when comparing this result to other researches available in the literature, it is noticed that many of them had their highest results when dealing with small sets (~ 30 subjects where ~10 are normal and ~20 are severe). The latter makes our results acceptable and the employed database was efficient when used for classification. The third classification version between normal and OSA patients on the other hand, achieved performance accuracy of 87.5% using three different features (VLF+VLF/HF +LF/HF) by the feedforward network and achieved 85% accuracy by the perceptron network with the three different features (LF+ HF+ VLF/HF). Finally, it can be observed that the highest achieved test set accuracy of 77.5% in the fourth classification version between normal, mild, moderate and severe OSA subjects was accomplished by the feedforward network using a combination of two statistical features of pNN20 and pNN30 respectively.

The representations in Fig. 14 demonstrate each type of network topology with different features as inputs for the classification versions corresponding to the best accuracies listed in Table 1.

For the third version, the perceptron network is sketched oxygenated blood although its performance accuracy was 85% and less than that of the Feedforward network 87.5%, in order to show the topology of the perceptron network.

## 9. CONCLUSION

Obstructive sleep apnea (OSA) is a common breathing-related sleep disorder affecting individuals of different age groups, genders and origins. It is characterized by short-duration cessations in breathing during sleep due to the collapse of the upper airway. OSA is associated with major co-morbidities such as cardiovascular diseases, arrhythmias, strokes, obesity, depression, certain types of hypertension and diabetes. The golden and reliable standard test for the detection of OSA is a polysomnographic sleep study. However, this test is time/labor-consuming, expensive and cumbersome. Analysis of a Heart Rate Variability (HRV) signal that is obtained from an Electrocardiograph (ECG) signal in time or frequency domain is an effective, non-invasive and promising method for the detection of OSA.

In this research, single perceptron, feedforward with back propagation and probabilistic artificial neural networks are investigated for their performance in classifying SQUH database subject's severity degree against four classification versions. The highest achieved accuracy of 95% was obtained when using VLF feature with the probabilistic neural network for normal vs. severe classification (version-1). The feedforward neural network achieved an accuracy of 95% as well when classifying normal versus mild OSA patients at a combination of LF and VLF/HF ratio features. In Version 3, the feedforward network achieves 87.5% accuracy using three features VLF and VLF/HF and LF/HF for normal vs. patient (including: mild, moderate and severe subjects in one group) classification. In the same version, the perceptron network achieved the highest performance accuracy of 85% using LF along with HF and VLF/HF ratio combination of features. Finally, for OSA severity degree classification (verion-4) statistical time-domain features provided the highest accuracy of 77.5% when using a combination of pNN20 and pNN30 features with the feedforward neural network.

The results are considered promising since the networks only used a maximum of three features to provide such results. Some of the limitations of this work include the ECG database size, neural networks training processing-time especially the feedforward network, feature dimensions (combinations of inputs). Future research recommendations are to be on investigating deep learning neural networks.

**Table 1.** Highest classification results of the networks at training scheme 1.

Network Type	Classification Version	Features	Specificity	Sensitivity	Accuracy
Probabilistic	Normal vs. Severe	VLF	100%	90%	95%
Feedforward	Normal vs. Mild	LF+ VLF/HF	90%	100%	95%
Perceptron	Normal vs. Patient	LF+ HF+VLF/HF	80%	86.67%	85%
Feedforward	Normal vs. Patients	VLF+VLF/HF +LF/HF	90%	86.67%	87.5%
Feedforward	Normal vs. Mild vs. Moderate vs. Severe	pNN20+pNN30	80%	76.67%	77.5%

### **CONFLICT OF INTEREST**

The authors declare no conflicts of interest.

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## A COMPARATIVE STUDY OF PHYSICAL PROPERTIES USING VARIOUS GRADES ASPHALT BINDER WITH DIFFERENT TYPE OF FIBERS

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**ABSTRACT:** For a long time, bitumen has been utilized as the essential material for asphalt pavement construction. The factors of increasing axle loads, increasing traffic movement, critical climate conditions and many forms failures in construction have steered many researchers to seek some methods to enhance the asphalt binder properties. Even though various types of modifiers have been utilized in strengthening asphalt concrete, fibers have attracted the most attention due to their high and desirable characteristics. It is realized that the good distribution of the modifier in asphalt binder can generate a strong network in the interior structure of the blend, causing bitumen mastic to be more coherent. In this study, a laboratory investigation of the rheological and physical properties of various grades of bitumen modified by two types of fibers was conducted. Three grades of asphalt were used in this study (60-70 penetration grade, 80-100 penetration grade and PG-76 grade) with two types of fibers with different percentages- Cellulose oil palm fiber (COPF) (0.15, 0.3, 0.45, 0.6, and 0.75%) by weight of asphalt and carbon fiber (0.75, 1.25, 1.75, 2.25, and 2.75%) by weight of asphalt. The results showed enhancement in physical performance of the modified bitumen in terms of the decrease in penetration values, as well as a rise in the softening point and viscosity values. The fibers' modified asphalt binders showed improved rheological properties and can raise the grade of asphalt depending on the base asphalt type.

Keywords: Carbon fiber; Cellulose oil palm fiber; Asphalt grade; Modified bitumen; Rheology.

## تأثير أنواع مختلفة من الألياف على الخصائص الفيزيائية لدرجات مختلفة من المثبتات الاسفلتية

سعدى عبد تايه \*، رنا عامر يوسف وقيس صاحب بني حسن

الملخص: منذ زمن بعيد كان ولا يزال القار يستخدم كمادة أساسية في الرصف بالأسفلت. و لقد دفعت عدة عوامل مثل ثقل الحمولة على المحور وزيادة حركة المرور والظروف المناخية القاسية والعديد من أشكال القصور في البناء العديد من الباحثين للسعي لإيجاد بعض الطرق لتقوية خصائص مواد تماسك الأسفلت. و على الرغم من استخدام أنواع مختلفة من المواد المحسنة في تقوية مواد تماسك الأسفلت، إلا أن الألياف جذبت أكبر قدر من الاهتمام بسبب خصائصها العالية والمرغوبة. وقد لاحظنا أن التوزيع المتساوي للمادة المضافة لتماسك الأسفلت ، إلا أن الألياف جذبت أكبر قدر من الاهتمام بسبب خصائصها العالية والمرغوبة. وقد لاحظنا أن التوزيع المتساوي للمادة المضافة لتماسك الأسفلت ، إلا أن الألياف جذبت شبكة قوية في البنية الداخلية للمزيج مما يؤدي إلى تماسك عجينة الاسفلت تماسكًا كبيرا. و قمنا في هذه ألدراسة بإجراء فحوصات مخبريه حول الخواص الحركية والفيزيائية لمختلف أنواع القار المعدل باستخدام نوعين من الألياف . حيث استخدمنا ثلاث درجات من الأسفلت إلى تشكل شبكة قوية في البنية الداخلية للمزيج مما يؤدي إلى تماسك عجينة الاسفلت تماسكًا كبيرا. و قمنا في هذه ألدراسة بإجراء فحوصات مخبريه حول الخواص الحرائية و المرغوبة. وقد لاحظنا أن التوزيع المتساوي للمادة المضافة للمزيج مما يؤدي إلى تماسك عجينة الاسفلت تماسكًا كبيرا. و قمنا في هذه ألدراسة بإجراء فحوصات مخبريه حول الخواص الحركية والفيزيائية لمختلف أنواع القار المعدل باستخدام نوعين من الألياف. حيث استخدمنا ثلاث درجة إختراق و درجة (0-70) مع نوعين من الألياف بنسب مختلفة. ألياف السليلوز من نخيل الزيت (0.5) (COPF) درجة إختراق ، 80-00 درجة إختراق ، 80-00 درجة (0.5 م 2.5 ، 2.5 ) ما وزن الأسفلت وألياف الكربون (0.5 ، 2.5 ، 2.5 ) ما 2.5 ، 2.5 ) ما وزن الأسفلت. أظهرت النتائج تحسن في الأداء الفيزيائي للسفل المعدل من حيث انخفاض قيم الاختراق ، وكذلك ار تفاع في ما وزن الأسفلت. أظهرت النتائج تحسن في الأداء الفيزيائي للسفلت المعدل من حيث انخفاض قيم الاختراق ، وكذلك ، 2.5 ، 2.5 ، 2.5 ) من وزن الأسفلت. أظهرت مواد تماسك مولف في الأداء الفيزيائي للسفلت المعدل من حيث انخفاض قيم الاختراق ، وكذلك ار تفاع في درجة المولي ال المولي المعلي مواد تماسك في الأوسات حيا معين من الألياف حيب من وي المال مولي مول ما معرا الموم مورن المولي مواد مولم مولي مولممات

*الكلمات المفتاحية:* ألياف الكربون؛ الياف السليلوز؛ نخيل الزيت؛ درجة الاسفلت؛ القار المعدل؛ الخواص الحركية.

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## 1. INTRODUCTION

The continuous increase in axle loads, increase in traffic movement, critical environment circumstances, and failures in pavement construction as well as the an enormous increase in asphalt binder prices have justified endeavors of many researchers to seek some methods to enhance bitumen properties by producing higher viscosity low-priced asphalt for pavement construction. There is an extensive range of applications of bitumen modifiers in road construction (Muniandy et al., 2008).

Regarding the rise in the consumption of bitumen all over the globe, several experiments were conducted to boost the characteristics of such product. Besides the developments in the field of designing concept and applying asphalt pavement, these trials have led to using more suitable materials required for the construction of asphalt pavement. Such materials may be used straight as modifiers to strengthen or to enhance the fundamental components of bitumen (Airey, 2004).

## 1.1 Carbon Fiber Modifier

Many types of additives were utilized to maintain the phase composition and advance the engineering properties and supply more strength to asphalt pavement (Ahmedzade, 2013). Lately, the appliance of fibers, as a method for asphalt modification, has concerned many researchers. This is due to their intrinsic compatibility with bitumen and exceptional mechanical characteristics, as carbon fibers could supply a good promise for asphalt modification. Modifying asphalt mastic with carbon fiber is expected to increase stiffness and permanent deformation resistance as well as improving fatigue characteristics of the mixture. The cold service temperature behavior of carbon fiber-modified asphalt mixtures was expected to be boosted as well due to the high tensile strength of the carbon fiber (Cleven, 2000).

Nejad *et al.* (2014) conducted research utilizing carbon fiber to strengthen asphalt concrete. The outcomes showed that the use of carbon fibers in the asphalt concrete can noticeably improve the mechanical properties that benefit all the related fields involved issues like construction and maintenance.

Physical properties of various types of fibers and their enhancing and strengthening influence on bitumen mastic properties were studied by many researchers (Ling *et al.*, 2014; Wu *et al.*, 2015). Their results revealed that fibers can shape a three-dimensional space composition in bitumen that may transfer and buffer the applied loads. This network can offer a decrease in drain-down of asphalt binder from the mixture throughout forming a thicker mastic coating for aggregate particles. Fibers can efficiently enhance the rutting resistance of asphalt mastic.

## 1.2 Cellulose Fiber Modifier

The introduction of cellulose fibers, from annually

renewable resources, is now a well-liked prevalence in the reinforcement of asphalt mastic. These offer advantages to the environment in relation to the degradability as well as utilization of materials from natural resources (Rout *et al.*, 2001). Cellulose is respected as the main framework element of the fiber structure. The benefits of natural lingo-cellulosic fibers over conventional strengthening materials are the satisfactory mechanical properties, low price, low density, suitable thermal properties, non-abrasivity, and improved energy salvage (Doan *et al.*, 2006; Abiola *et al.*, 2014).

There are some researches that has attempted to investigate the potential of cellulose fibers to bolster asphalt binder. Kumar *et al.* (2004) studied the modification of SMA with two types of fibers, jute and synthetic. Jute fiber SMA mixtures showed better permanent deformation and higher creep modulus than synthetic fibers SMA mixtures. Yang *et al.* (2006) investigated the resistance to deformation resistance of cellulose and polyester fibers modified bitumen. The achieved results displayed the significant improvement of the rut resistance. However, polyester fibers were relatively better regarding the rutting resistance than cellulose fibers samples do.

Some researchers evaluated the influence of date palm and textile fibers on open graded course mixture characteristics. The results have shown that the utilization of fibers in the asphalt mixture led to a considerable decrease in drain-down contrasted to other types of modified mixtures (Hassan and Al-Jabri, 2005; Sharma and Goyal, 2006). Muniandy and Huat (2010) used Cellulose fiber extracted from oil palm bunch to study the rheological performance of asphalt binders. The tests have shown the base binder (PG64-22) could be upgraded to PG70-22 grade as well as improving the fatigue performance of SMA mixture. The optimum cellulose fiber to be introduced to the base bitumen was found to be 0.6%. Another research conducted by Muniandy et al. (2008) dealt with the advantage of the Date Empty Fruit Bunch (DEFB) and Oil Palm trees to generate cellulose fiber to be used as additives in the bitumen. The outcomes showed that the used fibers improved the rheological properties of the asphalt mastic by increasing the PG grading two steps from PG58 to be PG70 at 0.3% oil palm cellulose fiber concentration.

Other researchers have attempted to study the influence of several cellulose fiber types and percentages on rheological and physical rheological properties of bitumen. The cellulose fiber modified bitumen displayed an increase in viscosity and softening point values as well as a reduction in penetration with increasing cellulose fiber content. The rheological tests showed an improvement in rutting resistance and fatigue cracking (Maniruzzaman et al., 2015a; Maniruzzaman et al., 2015b; Al-Otaibi et al., 2016; Bonica et al., 2016). According to the literature stated above, the

application of fibers and tire crumb rubber in asphalt may enhance the mechanical behavior represented by fatigue performance, strength, and Marshall Stability. However, for comparative purposes, the extent of the effect of each type of modifiers and their optimum proportion of adding them to the base asphalt cement have a big influence on the implementation of bitumen. So, the major objective of this study is to realize the best additive amongst them and the best percentage for strengthening bitumen mortar throughout a series of tests on the rheological and physical properties of the modified and unmodified bitumens. To this end, cellulose oil palm fiber, carbon fibers, and tire crumb rubber with different proportions by weight of the binder are added to three types of binders (80/100, 60/70, and PG75) to be tested and evaluated.

#### 2. MATERIALS AND METHODS

#### 2.1 Asphalt Binders

Traditionally the 80/100 binder is incapable of opposing the heavy weights from different trucks because it does not have a suitable fatigue and rutting performance. The use of modifiers such as crumb rubber and fibers may improve the service properties of the asphalt mastic by different mechanisms. Therefore, for the purpose of investigating the effect different additives on different grade asphalt binders, three grades of asphalt were used as base asphalt binders (80-100 and 60-70 penetration grade asphalts as well as PG-76 performance grade asphalt). The laboratory assessments were conducted in order to estimate the binders' properties for penetration, softening point, viscosity, specific gravity, and ductility. Table 1 shows the physical properties of the three base binders used in this study.

#### 2.2 Additives

Two types of additives were used in this study:

**2.2.1** Carbon Fiber: The physical properties of carbon fiber employed in this research are summarized in Table 2.

**2.2.2.** Cellulose Oil Palm Fiber (COPF): The oil palm bunches were collected from a plantation area then cut into small pieces. The cellulose oil palm fibers were made by chemical-R of pulping method. The oil palm fibers used are with a moisture content of about 5% and a cellulose content of at least 75% and a pH of 7±5%. The ash content is 3.5% and the density is about 1.5 g/cm<sup>3</sup>. The cellulose oil palm fiber size distributions are presented in Table 3.

#### 2.3 Preparation of modifier-asphalt mastic

Modified asphalt binder specimens were made according to standard techniques employed and

published in several journal researches. All the prepared samples were derived from the same neat asphalt binder sources.

Two kinds of fibers (cellulose oil palm fiber, and carbon fibers) were used as modifier. A high shear mixer was used to achieve an excellent distribution of fibers into the base binders with a revolution of about 1000 rpm. The fibers were discretely placed into the oven at 105°C for 24 h to make sure that the least moisture, and a 500 g sample of bitumen were put into a container and heated at 160°C for 30 min to soften it prior to blending. To explore the effect of fibers on bitumen mortar, various fiber proportions in the asphalt mortars were added. The cellulose fibers (COPF) contents used were (0.15, 0.3, 0.45, 0.6 and 0.75%) by weight of asphalt, while the carbon fiber contents used were (0.75, 1.25, 1.75, 2.25 & 2.75%) by weight of asphalt. Blending then continued at 160°C for 30 min to provide a homogeneous blend. In order to prevent the contrary influence of extreme heating, the temperature was cautiously observed using a thermometer with a thermocouple probe. After completion, the modified binder was extracted from the metal tins and separated into small containers enclosed by aluminum foil then kept for further testing at room temperature.

#### 3. RESULTS AND DISCUSSION

#### 3.1. Conventional tests

The traditional tests, like penetration (ASTM D5), softening point (ASTM D36) and viscosity (ASTM D4402), were first conducted for the neat binders and then for modified binders according to standards.

**3.1.1 Penetration test:** Figs. 1 and 2 show penetration test results for neat and modified asphalt binders. From this figure, it can be noticed that, with the increase in the percentage of modifiers, the value of penetration decreases. On the other hand, the figures show that cellulose fiber modified binders have higher values of penetration as compared with carbon fiber asphalt binder samples. The penetration value of the modified high-quality binder samples (PG76) did not change significantly.

Table 1. Physic	cal Properties	of Asphalt	Cement.
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Binder properties	Specification	80-100	PG-76	60-70
Penetration (25 °C, 100gm, 5 sec.) (0.1mm)	ASTM D5	84	33.5	63.5
Softening point (ring & ball) °C	ASTM D36	46.5	73.5	49
Viscosity at 135°C (cP)	ASTM D4402	297.45	1541.8	382.3
Ductility (25°C, 5cm/min).	ASTM D113	>100	>100	>100
Specific gravity at 25°C (g/cm <sup>3</sup> )	ASTM D70	1.036	1.03	1.04

A Comparative Study of Physical Properties Using Various Grades Asphalt Binder with Different Type of Fibers

Source	100% carbon fiber from Japan	Tensile strength 3.53 (GPa)
Yarn type	12K carbon fiber	Tensile Modulus 230 (GPa)
Chopped length (µm)	150	Carbon content $\geq 95$ (%)
Filament diameter (µm)	7.0	Density (g/cm3) 1.76

 Table 2. Physical Properties of Carbon Fiber.



Figure 3. Effect of Carbon Fiber Content on Softening Point for Asphalt Binders.

**Table 3**. Cellulose Oil Palm Fiber Size Distribution.

No.	Sieve size (µm)	% Passing
1	580	90
2	425	80
3	250	65
4	180	55
5	150	35
6	106	20

 
 Table 4.
 The Maximum Allowable Percentage of Additives to Each Base Binder Type.

Additive Type	Binder Type	Max. Additive %
	80-100	1.75
Carbone Fiber	60-70	1.35
	PG76	1.1
	80-100	0.28
Cellulose Fiber	60-70	0.23
	PG76	0.1



Figure 1. Effect of Carbon Fiber Content on Penetration for Asphalt Binders.



Figure 2. Effect of Cellulose Fiber Content on Penetration for Asphalt Binders.



Figure 4. Effect of Cellulose Fiber Content on Softening Point for Asphalt Binders.



Figure 5. Effect of Carbon Fiber Content on Viscosity for Asphalt Binders.

■ 80-100 ■ 60-70 ■ PG76



Figure 6. Effect of Cellulose Fiber Content on Viscosity for Asphalt Binders.

**3.1.2.** Softening point test: The outcomes of the softening point test for the three types of binders and two types of fibers are shown in Figures 3 and 4. It can be seen, as opposed to the penetration measurements, with increasing the fiber percentage, the softening point increased. There were somewhat significant changes in softening point values liable on the sort of the base binder. However, the softening point values for the high-quality asphalt binder (PG76) modified by

the two fiber types did not change significantly.

3.1.3. Viscosity tests: Figures 5 and 6 show the viscosity values of the control and modified bitumen. The viscosity values of the fiber modified bitumen are greater than the base bitumen. The more modifiers are introduced into the base bitumen, the greater viscosity values are gained. Some viscosity values of the fiber modified asphalt binders were higher than 3 Pa.s, and so didn't fulfill the requirement standard of Superpave<sup>™</sup>. Based on that, the recommended proportions to add each additive to each of the three base binders used in this study were estimated and presented in Table 4. The viscosity value is a crucial issue for transportation of bitumen. Excessively high value of viscosity is unfavorable when transporting bitumen using pipelines. Thus, over-viscous bitumens are inappropriate for use in pavement industry.

Based on the previous outcomes, the introduction of the two fibers into the base asphalt binders enhances the physical properties of the modified bitumen binders as indicated by the reduction in penetration value and increase in softening point, and an increase in the viscosity values, thus enhancing modified binder stiffness and increasing its capability to resist rutting deformation.

#### 3.2 Rheological properties for modified asphalt binder

The dynamic rheological characteristics of the cellulose modified asphalt mastic were determined utilizing a dynamic shear rheometer (DSR, HAAKE, Rheo Stress RS1, Phoenix) throughout an extensive extent of temperatures to illustrate both viscous and elastic behaviors of unaged modified binders. Two 25mm diameter parallel plates with a gap of 1mm were determined to do the DSR measurements. A constant sinusoidal stress of 0.12 kPa was directed at low strain to keep all the tests performed within the linear viscoelastic limit. The real strain and rotation were measured for determining viscoelastic factors, counting complex modulus ( $G^*$ ) and phase angle ( $\delta$ ). Furthermore, the rutting factor (G\*/sin\delta) was utilized to primarily measure the rutting resistance and stiffness of the asphalt mastics (Yi-qiu et al., 2010).

#### 3.2.1. Temperature sweep test at high temperature:

The fundamental rheological properties of the neat asphalt and modified bitumens, cellulose fibers (COPF) and carbon fiber by temperature sweep test at un-aged state using dynamic shear rheometers (DSR). A DSR oscillation temperature sweep test was performed at intermediate and high temperatures that ranged from 40 to  $82^{\circ}$ C with a 6-degree step to determine the change in binder performance as a function of temperature and 10 rad/s (1.59 Hz) frequency. A minimum value of 1.0 kPa for G\*/sin  $\delta$  is recommended to ensure that asphalt binder could resist well against permanent deformation at the designed performance grade temperature. The minimum limit for G\*/sin  $\delta$  is 1000 Pa for an unaged asphalt binder.

Figs. 7 and 12 present the relation between the rutting factor and temperature during temperature sweep test showing the failure temperatures for the two types of modified binders.

The cellulose modified bitumens have greater values of rutting factor than the control bitumens. Consistent with the specification, these modified bitumens have a reduced rutting susceptibility, especially at high service temperature contrasted to the base binders. From Table 5, it can be seen that the failure temperatures of the modified asphalt binders are greater than the base asphalt binders. As a result, the high-temperature grade (PG) for the base binders has been increased.

The addition of the two fiber types into the base bitumens caused them to be stiffer. Greater G\* and G\*/sino of these modified bitumens show that the bitumen could sustain additional energy when loading is directed, and similarly, they reveal that the modified binders have better resistance to rutting and fatigue cracking under intermediate and high service temperatures. The asphalt binders modified with carbon fiber mostly have similar resistance to rutting, which are clearly better than the control bitumens. At the same time, the base binder PG76 was the highest in terms of rutting resistance and stiffness followed by 60/70 and 80/100 binders respectively. So, the control binder source has a great impact on the rutting parameter values of various binders in this research. It can be evidently seen that the unmodified binders are categorized as PG58, PG64, and PG76, respectively. Coming back to the maximum failure percent of each additive to each control asphalt type according to the viscosity test results, the addition of 1.75 and 1.35% carbon fiber to 80-100 and 60-70 asphalt binders, respectively, had enhanced the PG grading two steps for 80-100 binder from PG58 to PG70, and enhanced PG grading one step from PG64 to PG70, while the effect of adding 1.1% of carbon fiber to PG76 binder did not change the PG grading significantly. The same trend is occurring with the addition of cellulose fiber to the three base binders, with slight differences. The addition of 0.28 and 0.23% cellulose fiber to 80-100 and 60-70 asphalt binders, respectively, had enhanced the PG grading two steps for 80-100 binder from PG58 to PG70 and enhanced PG grading one step from PG64 to PG70, while the effect of adding 0.1% of cellulose fiber to PG76 binder did not change the PG grading significantly.

## 4. CONCLUSION

In this paper, the effects of adding cellulose oil palm fiber, and carbon fiber to three asphalt binder types were studied. There are several conclusions derived from the results:

> • The two modifiers are well dispersed in the asphalt, due to the fact that no blocks or agglomerates can be seen after the



Figure 7. Temperature Sweep Test for 80-100 Asphalt Binder Modified with Carbon Fiber.



Figure 8. Temperature Sweep Test for 60-70 Asphalt Binder Modified with Carbon Fiber.



Figure 9. Temperature Sweep Test for PG76 Asphalt Binder Modified with Carbon Fiber.



Figure 10. Temperature Sweep Test for 80-100 Asphalt Binder Modified with Cellulose Fiber.



Figure 11. Temperature Sweep Test for 60-70 Asphalt Binder Modified with Cellulose Fiber.



Figure 12. Temperature Sweep Test for PG76 Asphalt Binder Modified with Cellulose Fiber.

|--|

	Carb	on Fibe	r %	that is received to be		10-0000	
Binder Type	0	0.75	1.25	1.75	2.25	2.75	
80-100	62.8	66.3	70.8	72.3	74	75.2	
60-70	69.5	70.6	72.2	73.8	75.5	76	
PG76	76.5	78	78.8	80.3	81.5	82.2	
	Cellulose Fiber %						
Binder Type	0	0.15	0.3	0.45	0.6	0.75	
80-100	62.8	66.5	71.5	73.3	75	77	
60-70	69.5	70.8	74	76	77.3	78	
PG76	76.5	77.5	79	79.2	80	80.2	

- The addition of carbon fiber and cellulose fibers to the asphalt binder improves the physical properties of the base asphalt binders as indicated by the decrease in penetration value and rise in softening point temperatures.
- The introduction of the two modifiers into base asphalt binders can considerably improve the viscosity of the bitumen, which is helpful to enhance the high temperature performance of asphalt binders. Cellulose and carbon fibers displayed very high viscosity values when added at high percentages to the base asphalt binders that exceed the specification requirement of 3 Pa.s at 135°C.
- Cellulose fibers have the most significant enhancement to viscosity and rutting factor of asphalt binders as compared with carbon fibers.
- Asphalt cement source may influence the behavior

of the modified mastic in different ways.

- The quality of the base asphalt binder is a crucial factor. Higher quality base asphalt binder showed little enhancements (PG76), whereas the lowest quality binders exhibit considerable enhancement in the conducted tests (80-100 and 60-70 binders).
- The DSR tests presented that fiber modified bitumen mastics have higher rutting parameters (G\*/sinδ) contrasted by with the base bitumen mastics employed in this research. Thus, fibers can significantly enhance asphalt mastic flow.

#### **CONFLICT OF INTEREST**

The authors declare no conflicts of interest.

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## POTENTIAL ENRICHMENT OF PHYSICAL PROPERTIES OF GYPSUM ADDING PALM TREE ASH AND SAW DUST

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**ABSTRACT:** Natural architecture uses locally produced materials to assure the strength of the construction elements. Palm tree is widely planted in the Gulf countries and its annual abounded residues constitutes a greater environmental burden of being neglected and rarely used or recycled in efficient ways. The paper discusses the potential changes in the physical properties of pure gypsum cubes when utilizing additives such as the ash and saw dust of palm tree residues. The additives are added in various proportions by weight. Four groups of different laboratory tests are conducted on four different groups of cubic samples made of pure gypsum powder, admixture of gypsum and palm tree ash, and admixture of gypsum and palm tree saw dust. Results showed that both sawdust and ash of palm tree have significantly enhanced the physical properties of gypsum cubes tested in this paper, weight, porosity, and compressive strength.

Keywords: Ash; Building Materials; Gypsum; Palm Tree; Properties; Saw Dust.

## فرص تحسين الخصائص الفيزيائية للجبس بإضافة رماد و نشارة أشجار النخيل

علي محمد القرني\* و محمد الحفناوي

الملخص: تستخدم الهندسة المعمارية الطبيعية المواد المنتجة محليا لضمان قوة عناصر البناء. وتُزرع شجرة النخيل على نطاق واسع في دول الخليج كما تشكل بقاياها السنوية الكبيرة عبنًا بيئيًّا كبير، ونادراً ما تُستعمل أو يُعاد تدويرها بطرق فعالة. تناقش هذه الورقة البحثية التغيرات المحتملة في الخواص الفيزيائية لمكعبات الجبس النقي عند استخدام مواد مضافة مثل الرماد ونشارة بقايا أشجار النخيل. تم خلط المواد المضافة بنسب مختلفة حسب الوزن، كما أجريت أربع مجموعات من الاختبارات المعملية على أربع مجموعات مختلفة من عينات مكعبة مصنوعة من مسحوق الجبس النقي (مجموعتين) ، ومزيج من الجبس ورماد شجرة النخيل (مجموعة) ، ومزيج من الجبس ونشارة شجرة النخيل (مجموعة). أظهرت النتائج أن كلا من نشارة الخشب ورماد شجرة النخيل قد عززتا بشكل كبير الخواص الفيزيائية لمكعبات الجبس المختبرة في هذه الورقة من حيث الوزن والمسامية وقوة الانضياط.

الكلمات المفتاحية: خصائص الجبس؛ رماد اشجار النجيل؛ مواد البناء؛ نشارة الخشب.

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## 1. INTRODUCTION

Gypsum is an important building material that has many commonly known uses, such as gypsum boards and paints. Gypsum is at the forefront of basic materials used in building and construction (EG 2017). It has many important characteristics such as: fire resistance, sound absorption, heat insulation, considerable bending strength, light weight, low cost, long life, fast solidification and ease of composition (Kami and Kami 1995). Current necessities for gypsum compounds for execution or mortaring comprise organized setting time, workability, sag resistance, compressive and flexural strength, ideal bond to bricks and concrete, waterproofing, and better These advances are thermal and acoustic. accomplished by the diligence of some chemical admixtures and mineral additives; among them are superplasticizers, water-soluble polymers and the admixtures responsible for retarding and airentraining (Arikan and Sobolev, 2002). In the last few years, considerable interest has been demonstrated in the investigation of date palm waste products as reinforcing and insulating materials to raise the mechanical properties of building materials. Consequently, much literature has been published on gypsum and the effects of some additives on its associated properties. This paper is conducted to investigate the properties of gypsum cubic blocks by adding the sawdust and ash separately to gypsum powder.

## 2. RELATED WORK

Danso el al. (2015) investigated the characteristics of mud blocks stabilized with leftover agricultural fibers, and performed the tests of density, shrinkage, water absorption, compressive and tensile strength, corrosion wearing. The results revealed and general enhancement in the physical, mechanical and durability properties of the blocks with the 0.5 wt. % fiber content. Sharma el al. (2016) presented an effort to develop the low durability of clay blocks by reinforcing it with the rural natural waste materials of Grewia Optiva and Pinus Roxburghii. The results showed an increase by 72 % to 68 % in the durability of the blocks reinforced with fibers of Grewia Optiva and Pinus Roxburghii. Sharma el al. (2015a) investigated improving the compressive strength of adobe using natural fibers of Grewia Optivia and Pinus Roxburghii. The reinforcement with these fibers resulted in an increase in the compressive strength by about 94-200 % and 73-137 % for Grewia Optivia and Pinus Roxburghii respectively. Sharma el al. (2015b) investigated enhancing soil sustainability and stabilizing it with natural fibers of Pinus roxburghii and Grewia optiva. Results revealed an increase in the compressive strength by 131-145 % when adding fiber P. roxburghii and 225-235 % when adding fiber G. optiva for the cubical and cylindrical specimens

respectively.

Zak *el al.* (2016) presented an experiment to enhance the compressive strength of earth bricks using different admixtures of earth, cement, gypsum, hemp and flax fibers. The results indicated that the impacts of adding fibers hemp and flax is very limited on the compressive strength. Cement and gypsum reduced the binding force of the clay and highly decreased strength.

Villamizar *el al.* (2012) investigated the effect of adding coal-ash and cassava skins on the properties of mud blocks. Results showed that stabilizing earth blocks with coal-ash improved the compressive and bending properties when added as less than or equal to 5%.

Ahmed *el al.* (1988) investigated the properties of date palm frond and concluded that tensile strength of the stalk walls is double that of the cores. The elasticity modulus of stalks is between 10 to 30 KN/mm2.

Arikan and Sobolev (2002) studied the effect of different admixtures on the compressive strength consistency. The additive admixtures are a superplasticizer, a water-soluble polymer, a retarding, and an air-entraining. The results proved that admixtures slightly increased the setting time, whereas, the application of a retarding admixture allowed control of a setting time of 1 to 3 hours.

Li *el al.* (2003) stated that the addition of cotton stalk to gypsum had a substantial impact on its mechanical properties. Cotton stalk fiber is treated with emulsion to increase its combination with gypsum. The results illustrated that, although the composite of gypsum had lower strength, its insulation and fire resistance properties had improved.

On the other hand, Wu (2004) studied the shear performance of gypsum panels when being longitudinally reinforced with glass fiber. The attained results showed that persistence of the longitudinal reinforcement had no significant issue on the shear strength, whereas it caused a small impression on their early stiffness, stiffness degradation, ductility ratio, and energy dissipation capacity.

Abu-Sharkh and Hamid (2004) studied the properties of a composite material produced by mixing palm leaves, polypropylene, and stabilizers. The results proved that uncompatibilized specimens are more stable than compatibilized ones. They justified their results that maleate polypropylene has low stability.

Kriker *el al.* (2005) tested the properties of palm date fiber reinforced concrete. It is found that increasing percentage and length of fiber in curing improved the flexural strength and the toughness coefficient and reduced the compressive strength.

Turgut and Algin (2007) mixed limestone powder and wood sawdust. The properties of the obtained material such as weight, water absorption, compressive strength, flexural strength and ultrasonic velocity satisfied the appropriate international standards. Therefore, the product had potentials for uses for walls, ceiling panels, sound panels, wooden board substitute, and a cheap alternate to concrete blocks.

Liangyuan and Zongli (2007) debated the features inducing the performance of gypsum-based composite material such as water/cement ratio, and retarder quantities. It is concluded that the strength and coefficient of softness increased compared to gypsum. Portland cement quickened the thickening and improved the strength and softness coefficient of composite, but over dosage caused the fall of these coefficients.

Jia-yan *el al.* (2008) studied the connection between wood-gypsum ratio and water gypsum ratio and explored the effects of temperature on hydration process and the gypsum morphology. It is concluded that both Water-gypsum ratio and wood-gypsum ratio are crucial to improve the properties of the boards in 40°C and to shorten the hydration time and strengthen the boards.

Okunade (2008) examined the impacts of adding sawdust and wood ash admixtures to clay mix to produce bricks. The results showed that the key influence of the sawdust admixture is reducing the dry density of the brick. Furthermore, the wood ash admixture attained denser products with higher compressive strength.

Brencis *el al.* (2001) produced an energy saving material made of foam gypsum and fibrous hemp in which sound absorption coefficient increased. It is also found that short fibers' reinforcement increased the density of foam gypsum, while long fibers decreased it.

Hai Alami (2013) studied the properties of clay mixture with desert solid wastes such as fronds and pits. It is proved that crushed pits expressively improved the strength of clay bricks adding more 85 % toughness, and 15 % strength.

Zhou *el al.* (2013) studied the properties of gypsum-based composites produced by mixing the powder of gypsum with latex and Polyvinyl Acetate (PVA). Release time, density and impact strength are investigated. It is proved that PVA contributed to delaying of time release and increased impact strength. On the other side, sawdust reduced the density.

Sari (2014) compared between two mixtures of polyethylene glycol with gypsum and clay in regard to low temperature-thermal energy storage. It is proved that compatibility between components in regard to capillary and surface tension enabled the composite for passive thermal energy storage applications in buildings.

Dai and Fan (2015a) developed a complex from gypsum and sawdust with a water-based epoxy spray. It is proved that sawdust water increased the flexural and compressive strength of gypsum by 10% and 7% respectively. Furthermore, analysis with optical microscopy confirmed that decrease of water uptake enlarged the gypsum covering ratio from 42 % to 68 %.

Dai and Fan (2015b) studied the mechanical properties

of a Bio-composite of wood sawdust and Gypsum. It is concluded that the saw dust ratio of 20% gained better flexural and compressive strengths compared with the 30% ratio.

Algarny *el al.* (2016) surveyed the feasibility of adding palm tree sawdust to gypsum powder to produce blocks for indoor usage. It is resolved that the shrinkage value, weight, and porosity value are significantly reduced by increasing the ratio of sawdust to gypsum powder, whereas compressive strength is increased at the same time.

## 3. MATERIALS AND METHODS

The materials used are pure gypsum powder, palm tree saw dust, and the ashes resulting from the burnt saw dust of the local palm tree fronds in the Eastern Province of the Kingdom of Saudi Arabia. Different mixing ratios of ash or saw dust to gypsum are added to produce gypsum cubes of  $5 \times 5 \times 5$  cm, Table 1. Three different laboratory tests are conducted at the same time for every two groups of the samples: weight, porosity, and compression strength. Humboldt Mortar Laboratory Mixer of 5L, which supports the ASTM C305 standard, is used. It is a bench top type that provides two mixing speeds of 140±5 rpm and  $285\pm10$  rpm. It has the dimensions of  $235\times396\times568$ mm. Materials described are mixed together in the dry state. Then, water is added and mixed until the mixture became homogeneous before conducting the tests. Mixing time is not to exceed one minute, so as not to get water evaporates. Each test is equipped with a number of 25 specimens, at a rate of 5 specimens for each mixing ratio. The result recorded in the test is the average value of the 5 specimens of each mixing ratio. The total number of the tested specimens are 150 specimens. Unlike a number of other specimens that are excluded for defects during the casting or the processing of the test raising, the number of specimens that are actually consumed to more than 200 specimens. In the weight test, the specimens are casted in their molds and left for 24 hours. Then, they are taken out and weighed by a digital scale. The arithmetic average is calculated for each mixing ratio using the recorded weights of 5 different specimens. The results are shown in Fig. 1. In order to conduct the porosity test, the specimens are undergone the following steps:

- 1. The dry weights of the specimens are measured by a digital scale.
- 2. The specimens are immersed in water for 24 hours.
- 3. The wet weights of the specimens are measured by a digital scale.
- 4. The following formula is applied to calculate the value of porosity in the specimen (ASTM 2000):

$$P,\% = [(W - D)/V] \times 100$$
(1)

P: Porosity	W: Saturated weight
D: Dry weight	V: Specimen Dry Volume

The results are shown in Fig. 3.

In the compressive strength test, for each admixture, five cubic specimens from each mixing ratio are prepared in a total of 50 specimens required for the test. The specimens are compressed in the compression strength instrument and the results are shown in Fig.4.

# 4. RESULTS AND DISCUSSION 4.1 Weight Test

As shown in Fig. 1, adding ash or sawdust reduced the specimens' weights. Similar results have been reported by the researchers Algarny *el al.* (2016), Li *el al.* (2003), and Arikan and Sobolev (2002). The addition of saw dust reduced the weight of the specimen more effectively than adding ash, as shown in the values of R2 for the trend lines of both results. It can be justified as the saw dust has less density and less weight for a specific volume compared to the ash. Taking into account the constant volume of the cubic specimen tested, adding more saw dust reduces more weight of the specimen than adding more ash.

Results indicate that the maximum weight reductions are 3.62% and 4.9% of the control specimen for ash and saw dust respectively at 6% mixing ratio. So, saw dust-gypsum admixtures would have more benefits for the construction industry and reduce the internal loads and stresses on the structural and construction elements of the buildings (Fig. 2).

#### 4.2 Porosity Test

As shown in Fig. 3, the addition of ash and sawdust significantly reduced the porosity value of the specimens. Lower porosity is recorded in higher mixing ratios. The result is consistent with the work reported by Algarny el al. (2016), and Okunade (2008). The porosity values are reduced by 4.80 % and 3.77 % in the 6 % admixtures of gypsum-ash and gypsum-sawdust respectively compared to the control specimens. Sawdust has a limited impact on decreasing the porosity of gypsum cubes compared to the ash, which might be because sawdust has a lower density and bigger particle size compared to ash. Therefore, for a specific weight of ash or saw dust added to the gypsum powder, sawdust has a relatively higher volume than ash leading to keeping more gaps between the particles of the gypsum powder compared to the case of adding ash which has a compacted higher density compacted form that introduces smaller quantities of gaps among the gypsum particles. So, gypsum-ash cubes are more convenient to applications that need less porosity compared to sawdust-gypsum cubes that suits the need for more porous materials.

Table 1. Miz	king rat	tios.	
Gypsum (gm)	Ash	/ Sawdust	Water (gm)
	%	(gm)	
3000	0	0	1950
	1	30	(65 % of Gypsum
	2	60	weight)
	3	90	
	6	180	







Figure. 2. Relationship between the weight loss of the specimens and the applied mixing ratios of the additives. Weight loss is directly proportional to mixing ratios.



Figure 3. Relationship between the porosity of the specimens and the applied mixing ratios of the additives.



Figure. 4. Relationship between the fracture loads of the specimens and the applied mixing ratios of the additives. Fracture load is directly proportional to mixing ratios.

#### 4.3 Compressive Strength Test

The results in Fig. 4 show that both the sawdust and ash have significantly increased the compression strength of the specimens and improved their failure resistance, which is consistent with the work reported by Algarny et al. (2016), Arikan and Sobolev (2002), and Okunade (2008). The resistance to fracture has increased by125.83 % and 62.9 % for the specimens of 6 % mixing ratio of sawdust and ash content respectively compared to the control specimen which is of pure gypsum. This result is tremendously important and can divert the use of gypsum in new building and construction applications due to its increased resistance to fracture in compression. Adding sawdust to the gypsum cubes will raise its compressive strength to almost double the value it will get if adding ash instead. The fibers content in the sawdust might be responsible for providing stronger bonds among the gypsum particles compared the case of adding ash which has already lost its fiber during the burning process.

The justification of the previous results can be explained as palm contains Wax, Phenol compounds and Pectin (Al-Dosary 2009), which have chemical properties to enhance the physical properties of gypsum powder. Wax solidifies at normal temperature and melts in heat (EB 2019a). When adding water, the wax melts from the emitted heat and spreads within the gypsum particles, then the wax hardens increasing the cohesion of the admixture blocking the pores of gypsum, and reducing the porosity value of the cubes, while increasing its strength. On the other hand, Pectin material gives the rigidity to the plant (Al-Dosary 2009), and when added to gypsum it increases its hardness and compression strength.

In addition, Phenol is soluble in water (EB 2019b), and it interacts with gypsum powder to produce salty water and stimulate the interaction between the granulated particles of gypsum and sawdust and ash of palm granules reducing porosity as well.

### 5. CONCLUSION

Both sawdust and ash of palm tree have significantly enhanced the physical properties of gypsum cubes tested in this paper. Both induced weight loss but sawdust achieved more reduction. The weight of gypsum specimen is inversely proportional to the amount of sawdust or ash content; as lower weights are recorded with higher content of the additives in the specimen. In line with the previous result, porosity ratio in the specimen of gypsum cubes is inversely proportional to the amount of ash or sawdust. Both additives reduced the porosity of gypsum cubes, but ash has a broader reduction. On contrary, compressive strength of the samples of gypsum cubes is directly proportional to the amount of ash or sawdust, where higher values of compressive strength are recorded in higher ratios of any of them in the specimen. The compressive strength the cubes gain when adding sawdust is almost double of that gained when adding the ash. The overall findings are encouraging for promoting the use of leftover palm tree residues to enhance the physical properties of some building and finishing materials which leads to adding an economic value and securing more environmental protection against soil and air pollution.

## **CONFLICT OF INTEREST**

The authors declare no conflicts of interest.

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## Probing the Effect of Gaseous Hydrocarbon Precursors on the Adsorptive Efficiency of Synthesized Carbon-Based Nanomaterials

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**ABSTRACT:** The present work investigates the effect of the type of carbon precursor on the adsorptive proficiency of as-prepared carbon nanomaterials (CNMs) for the removal of methylene blue dye (MB) from aqueous media. A comparison study was applied to assess the growth of CNMs from the decomposition of methane (CNMY1) and acetylene (CNMY2) using response surface methodology with central composite design (RSM/CCD). The produced nanomaterials were characterized using FESEM, EDX, TEM, BET surface area, Raman, TGA, FTIR, and zeta potential. The as-prepared adsorbent displayed different morphologies and under the experimental conditions, 10 mg of CNMY1 and CNMY2 was responsible for 97.7 % and 96.80% removal of dye. The maximum adsorptive uptake predicted by Langmuir isotherm was about 250 and 174 mg/g for CNMY1 and CNMY2, respectively. The as-synthesized carbon nanomaterial in this study could be explored as a great potential candidate for dye-bearing wastewater treatment.

Keywords: Adsorption; Carbon nanomaterials; Chemical vapor deposition; Dyes; Response surface methodology.

## التحقق من تأثير السلائف الهيدروكربونية الغازية على الكفاءة الإدمصاصية للمواد النانوية المستخلصة من الكربون

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الملخص: في هذه الورقة البحثية، تم تصنيع انابيب الكربون النانوية من نوعين مختلفين من الغازات الهيدركربونية (الميثان والاستيلين) بواسطة مفاعل كيميائي تحليلي تبخيري وعامل مساعد ثابت. وقد استخدم البرنامج الحاسوبي (الميثان والاستيلين) بواسطة مفاعل كيميائي تحليلي تبخيري وعامل مساعد ثابت. وقد استخدم البرنامج الحاسوبي والتي تتضمن درجة حرارة التفاعل، زمن التفاعل، ونسبة خلط الغازات المستعملة على ان تتحقق في المنتوج أفضل كمية والتي تتضمن درجة حرارة التفاعل، زمن التفاعل، ونسبة خلط الغازات المستعملة على ان تتحقق في المنتوج أفضل كمية والتي تتضمن درجة حرارة التفاعل، زمن التفاعل، ونسبة خلط الغازات المستعملة على ان تتحقق في المنتوج أفضل كمية مو مع التي تتضمن درجة حرارة التفاعل، زمن التفاعل، ونسبة خلط الغازات المستعملة على ان تتحقق في المنتوج أفضل كمية (FEESM مع افضل قدرة على از الة صبغة الميثيل الازرق من الماء. لقد فحصت خواص المادة الناتجة باستخدام TGA، والتي وجهد الزيتا المستعملة على المادة الناتجة باستخدام FTIR، ووجهد الزيتا الملك المرابي والاستلين لي والاستين لي والاستين لي المردة على از الة صبغة الميثيل الازرق من الماء. لقد فحصت خواص المادة الناتجة باستخدام GAX، والتي والاستلين لي وجهد الزيتا المين الذري والاتين والاستين والاستلين والاستلين والاستلين لي والد على أولان والاستين لي والدو على از الة الملوث بنسبة 20.5 و 10 ملغرام من المادة الممتزة الناتجة من تفكك الميثان والاستلين لي القدرة على از الة الملوث بنسبة 20.5 و 20.6 % على الترتيب. ووجد ان الايزويثرم حسب الميثان والاستلين لي القدرة على از الة الملوث بنسبة 20.5 و 20.6 % على الترتيب. ووجد ان الايزويثرم حسب الميثان والاستلين لي القدرة على از الله الملوث بنسبة 20.5 و 20.6 % على الترتيب. ووجد ان الايزويثرم حسب الميثان والاستلين والاستلين والاستلين والاستلين والاستلين والاستلين والاستلين لي القدرة على از اله الملوث بنسبة 20.5 و 20.6 % على الترتيب. ووجد ان الايزويثرم حسب الميثان والاستلين والاستلين والاستران والاستران المادة النادية التي والاستلين والاستريب. ووجد المامزة الماميثان والاستلين والاستريب. والامتزان والاستريبة والاستريب المادة المامية، وولامي والامتزان والاميزان والاستريب. وولام مالمادة الميثان والاستريب والامر والالماريب. والامالوية التي ماليي والاميزيب. والامري

الكلمات المفتاحية: الامتزاز؛ انابيب كاربونية نانوية؛ مفاعل تبخيري ذو عامل مساعد؛ الاصباغ الملوثة.

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## 1. INTRODUCTION

Carbon nanomaterials (CNMs) have been intensively investigated for environmental applications due to their outstanding properties. They have enlightened different prospects as attractive sorbents for the removal of undesirable pollutants from water. Their high surface area, hybridized  $sp^2$  carbon cluster and optimal binding efficiency enable strong adsorption capacity for organic contaminants (Hu *et al.* 2019; Sun *et al.* 2016).

Among the various techniques being used to synthesize CNMs, the chemical vapor deposition (CVD) is considered as the most promising technique, due to its versatility and suitability for large scale production. In CVD process and under tunable growth conditions, a statistical study should be conducted to achieve an adequate evaluation of the optimal growth conditions for CNMs, such as the utilized catalyst, carbon precursor type and flow rate, growth temperature and reaction time (Alayan *et al.* 2019; Gromov *et al.* 2016).

The agglomeration and ineffective recovery of CNMs are considered significant limitations which will confine their application in environmental remediation. Thus, it is essential to develop innovative adsorbents that can overcome the current challenges. Possible techniques proposed to resolve drawbacks include centrifugation these and attachment of magnetic nanoparticles. Nevertheless, both options will crucially add cost and complexity to the adsorption process. Accordingly, it is believed that growing carbon nanostructures on micro-scaled carbon support will enlighten the production of promising alternatives to the conventional adsorbents (Meshot et al. 2017).

Industrial effluents accept worrying quantities of colored pigments which impose harmful effects on the human health and surrounding ecosystems. Methylene blue (MB) contaminant has a potential danger of bioaccumulation due to its stability and low biodegradability, hence, efficient removal techniques of practical significance is imperative to remove it from industrial wastewaters before it is released to the environment. Of all treatment methods, adsorption based process has been widely implemented since it is simple, bear large volume of wastewater and devoid of utilizing organic solvents (Elsagh *et al.* 2017; Siddiqui *et al.* 2019).

In this work, we aim to assess the effect of hydrocarbon source being supplied in CVD reactor on the adsorption capacity of the as-prepared CNMs. Unlike previous studies that usually employ metallic substrates in the growth process of CNMs we fabricated our carbon nanomaterial by incorporating powder activated carbon (PAC) as analogues of non-carbon supports to boost the synergetic effect of hybridized CNMs to enhance their adsorptive affinity (Alayan *et al.* 2017). In this context, PAC was employed as a catalyst substrate to motivate the growth of CNMs from methane and acetylene

decomposition. Response surface methodology with central composite design (RSM-CCD) technique was applied to optimize the preparation parameters followed by characterizing the as-prepared nanomaterial using EDX, BET, FESEM, TEM, Raman spectroscopy, TGA, FTIR, zeta potential and Goniometer. Later, the adsorption performance of the fabricated hierarchical CNMs was investigated for the removal of MB dye from water samples.

## 2. MATERIALS AND METHODS

## 2.1 Materials

Methylene blue, powder activated carbon, nickel (II) nitrate hexahydrate Ni  $(NO_3)_2 \cdot 6H_2O$ , were purchased from Sigma-Aldrich. Methane CH<sub>4</sub>, acetylene C<sub>2</sub>H<sub>2</sub>, hydrogen H<sub>2</sub>, nitrogen (N<sub>2</sub>), were supplied in analytical grade and used without further purification.

## 2.2 Synthesis of CNM/PAC using CVD

A mixture of 2 g of dried PAC and 5 mL catalyst solution of nickel (II) nitrate hexahydrate (1.0 wt %) was sonicated at 60°C for 1 h. Then the Niimpregnated PAC was dried at 100°C overnight. The dried and grinded Ni/PAC samples were exposed to two sequenced stages of thermal treatment in CVD reactor including calcination and reduction at 350 and 600 °C for 2 and 1 h under 200 mL/min of N<sub>2</sub> and H<sub>2</sub>. The growth of hierarchal CNM/PAC was achieved by shuffling a ceramic boat of the Ni/PAC into the reaction tube of CVD reactor. The growth process was conducted with the following conditions: CH<sub>4</sub> and H<sub>2</sub> were introduced to the CVD reactor at a ratio range of 1-4 within a temperature range of 750-1000 °C for 20 -60 min. Then the reactor was cooled and the produced CNMs were collected. The second hierarchal CNM/PAC was synthesized from the decomposition of C<sub>2</sub>H<sub>2</sub> following the same experimental procedure. However, in this case the growth reaction was conducted within the temperature range 550-750°C. The yield of CNMs (CNMY) was calculated using equation (1):

$$\text{Yield} = \frac{M_2 - M_1}{M_1} \tag{1}$$

where  $M_2$  and  $M_1$  are the weight of the sample after and before reaction, respectively.

## 2.3 Experimental Design and Optimization

A design of experiment using CCD was employed to investigate how to obtain optimal growth parameters namely, hydrothermal temperature, reaction time and input gas ratio. The suggested regression model and the relevant statistical parameters were evaluated by the analysis of variance (ANOVA). Additionally, for optimization sake, maximizing the growth of CNMs was our response criterion. Accordingly, tridimensional plots were constructed to demonstrate the interaction between the underworking growth parameters.

#### 2.4 Characterization

The PAC, Ni/PAC, and CNMs were dispersed onto copper grids to examine their surface morphologies using SEM equipped with Energy Dispersive X-Ray Spectroscopy and Transmission Electron Microscopy. Raman spectra and thermogravimetric analysis of CNMs were developed. The BET method was carried out to determine the surface area of the prepared nanomaterials. The FTIR spectra over the range 4000 to 400 cm<sup>-1</sup> was used to analyze the surface changes. Surface charge analysis was investigated using the zeta potential measurements of a 0.01 wt % suspensions by Zetasizer. Finally, to debate the hydrophobicity issue, the goniometer was used to assess the surface hydrophobicity in terms of its contact angle.

#### 2.5 Measurements of Dye Performance

The removal of MB on the as-prepared CNMs produced from the decomposition of  $CH_4$  and  $C_2H_2$  were carried in a batch mode as follow: a fixed amount of each adsorbent (10 mg) was agitated for 30 min at room temperature into the dye solution (50 ml) at initial concentration of 50 mg/L and pH 11. At the end of the adsorption experiments, the dye solution was centrifuged and the persisted concentration was determined using UV-vis spectroscopic at maximum wavelength (665 nm). The removal efficiency and the adsorbent uptake (mg/g) were calculated by the following relationships:

Removal Efficiency (%) = 
$$\frac{C_o - C_e}{C_o} \times 100$$
 (2)

$$Uptake = \frac{C_o - C_e}{W} \times V$$
 (3)

where Co and Ce (mg /l) are the initial and equilibrium dye concentrations, respectively, V (L) is the volume the dye solution and W (g) is the amount of the adsorbent. The adsorption efficiency can be demonstrated via the analysis of experimental data to estimate the best fitted kinetic models and adsorption isotherms (Chen and Bai 2013). Kinetics of adsorption of MB on as-prepared CNM were investigated using both the pseudo-first-order and pseudo-second-order models. In addition, Langmuir and Freundlich isotherms were applied to study the sorbent-sorbate interactions.

#### 3. RESULTS AND DISCUSSION

#### 3.1 Model Establishment and Statistical Analysis

The growth temperature (A), growth time (B) and input gases flow ratio;  $(H_2/CH_4)$  or  $(H_2/C_2H_2)$  (C) were optimized for CNM/PAC synthesis using 2-level

CCD with one central point by the RSM/CCD method. The DOE software suggested conditions covered the growth temperature of 750 -1000 °C, 550 - 750 °C for CH<sub>4</sub> and  $C_2H_2$  decomposition, respectively. Also, for both hydrocarbon precursor, the reaction time and gases ratio were varied under the range of 20 - 60 min, 1.0 - 4.0. The optimizing criterions were devoted to maximizing the yield percentage of CNMY1 and CNMY2 for CH<sub>4</sub> and  $C_2H_2$ , respectively, while the independent parameters were kept in prescribed ranges.

The ANOVA summarized in Table 1 shows that the major determinants for CNMY1 obtained from CH<sub>4</sub> decomposition are the growth temperature (A), the interaction of the growth temperature and the gas ratio (AC) and the second-order effect of growth temperature (B<sup>2</sup>). The high model F-value and the low p- values < 0.05 of these confirm their significance effect. The high correlation coefficient R<sup>2</sup> value for CNMY1 and CNMY2 (> 0.98) suggest that the

Table 1. ANOVA results for (CNMY1).

*Source	Sum of Square	df	Mean Square	F-Value	<i>p</i> -value
Model	2.71	10	0.27	210.54	0.0005
А	1.18	1	1.18	914.87	< 0.0001
В	0.074	1	0.074	57.22	0.0048
С	0.068	1	0.068	52.94	0.0054
AB	0.11	1	0.11	84.02	0.0027
AC	0.23	1	0.23	178.35	0.0009
BC	0.0087	1	0.0087	6.76	0.0804
A <sup>2</sup>	0.009	1	0.009	69.48	< 0.0036
$B^2$	0.043	1	0.043	33.05	0.0105
$C^2$	0.0011	1	0.0011	0.89	0.4158
$A^2C$	0.048	1	0.048	37.40	0.0088
	R- Square	d 0.99	98	Std.Dev. 0.	0359
	Adj. R-Sq	uared	0.994	C.V. % 1.3	5
	Pred. R-S	quare	d 0.938	precision 4	6.61
*A (°C): T	*A (°C): Temperature, B (min): Time,				
C (feed ga	s flow ratio	): H <sub>2</sub> /	CH <sub>4</sub>		

 Table 2. ANOVA results for CNMY2.

	Sum of		Mean		
Source	Sull of	df	Squar	F-Value	<i>p</i> -value
	Square		e		
Model	0.692	8	0.0865	101	0.00986
А	0.0176	1	0.0176	20.4	0.0456
В	0.0522	1	0.0522	60.8	0.0160
С	0.0334	1	0.0334	38.9	0.0247
AB	0.0654	1	0.0654	76.1	0.0129
AC	0.0921	1	0.0921	107.0	0.00920
BC	0.231	1	0.231	269.0.	0.0037(
A2	0.0197	1	0.0197	23.0	0.0409
A2 B	0.0428	1	0.0428	49.9	0.0195
	R- Square	ed 0.99	98	Std. Dev.0	.0293
	Adj. R-So	quared	0.988	C.V. % 0.0	504
	Pred. R-S	quareo	d 0.929	precision 2	26.4
*A (°C): Temperature, B (min): Time,					
C (feed	gas flow ra	tio): H	$I_2/C_2H_2$		



Figure 1. Predicted vs. actual values; (a) CNMY1, (b) CNMY2.

 
 Table 3. The optimum conditions suggested by DOE for CNMY1.

No.	Α	В	С	CNMY1%	Desirability
1	950	60.0	1.00	36.7	0.989 🗸
2	950	59.8	1.00	36.5	0.986
3	950	60.0	1.00	36.5	0.986
4	950	60.0	1.03	36.4	0.984
5	950	59.7	1.00	36.3	0.982
6	950	60.0	1.13	35.7	0.970
7	950	60.0	1.22	35.1	0.959
8	950	60.0	1.00	33.4	0.927
9	950	60.0	1.52	33.0	0.917
10	950	20.0	1.00	23.0	0.674

 Table 4. The optimum conditions suggested by DOE for

 CNMV2

	C1 117.	112.			
No.	Α	В	С	CNMY2 %	Desirability
1	560	38.0	1.00	154.47	0.919 🗸
2	561	38.0	1.00	152.9	0.916
3	560	38.5	1.00	154.47	0.914
4	560	38.0	1.12	152.9	0.913
5	561	38.4	1.00	154.47	0.912

obtained model offers a successful correlation between the growth variables and good estimation of the responses which is confirmed by the plots of their predicted values against the actual values presented in Fig. 1a and 1b. Furthermore, the statistical results in Table 2 support the significance of the suggested model for CNMY2 from  $C_2H_2$  pyrolysis for the linear terms (A), (B), and (C). The interaction effect of (AC) and (BC) displayed p-values of 0.009 and 0.0036, respectively indicating their strong impact over the studied range as well. The regression equation for CNMY1 and CNMY2 in terms of their actual values are given by equations (4) and (5):

$$Ln CNMY1 = -11.4 + 0.0304 A + 0.0789 B + 11.0 C + 5.79 \times 10^{-5} AB - 0.0251 AC - 0.0011 BC - 1.45 \times 10^{-5} A^2 + 0.00046 B^2 + 0.0128 C^2 + 1.41 \times 10^{-5} A^2 C$$
(4)

 $Ln CNMY2 = 26.0 - 0.0637A - 0.385 B - 0.28 + 0.00121 AB + 0.000715 AC - 0.00566BC + 4.59 \times 10^{-5}A^2 - 8.96 \times 10^{-7}A^2B$ (5)

#### 3.1.1 Response Surface Methodology

The effect of the substantial factors and the feature of the obtained response surface in the experiment were examined by using RSM/CCD. The interaction effects of the process parameters on CNMY1 and CNMY2 based on the empirical model were illustrated by RSM plots. Fig. 2 displayed an outstanding increase in CNMY1 with increasing the reaction temperature from 750 to 950°C, implying the favor of high reaction temperature for the growth of CNMs Fig. 2a. There was no significant improvement for CNMY1 with the increase in (H2/CH4), since high concentration of H2 will motivate the backward reaction thus reducing the yield of CNMY1 Fig. 2b. Also, the CNMY1 improved with increasing growth time and then dropped as the reaction proceeds Fig. 2c. This decrease in the yield of the CNMs at long reaction period is attributed to the catalyst deactivation due to the formation of amorphous carbon. In Fig. 3a, increasing reaction temperature from 550 to 650°C has a distinguished improvement on the throughput of CNMY2 at fixed H2/C2H2 (1.0) which is in agreement with the role of PAC substrate in minimizing the deactivation of impregnated catalyst even at high growth temperatures (Tao and Crozier 2016). However, an increase in CNMY2 for low reaction temperatures and H2/C2H2 at fixed reaction time is observed in Fig. 3b, and the high temperature accompanying with high gas flow ratio was unfavorable for CNMY2. This might be since the high H2 content will accelerate the reduction of metal oxides which in turn encourages the deposition of pyrolytic carbons on the catalytic sites. Moreover, it can be noticed from Fig. 3c that at short reaction time the yield of CNNMY2 was high and almost the same at H2/C2H2 of 1.0 and 4.0 which indicates the insignificance effect of the gas ratio. This observation motivates the production of CNMs at small reaction periods to get high yield and prevent catalyst deactivation as well (Shen et al. 2016).



Figure 2. Response surface illustration of the interaction effects on CNMY1: (a) growth temperature and time, (b) growth temperature and gas ratio and (c) time and gas ratio.



Figure 3. Response surface illustration of interaction effects on CNMY2: (a) growth temperature and time, (b) growth temperature and gas ratio and (c) time and gas ratio.





Figure 4. (a) FESEM, (b) TEM and (c) EDX images for Ni/PAC before the growth reaction.



Figure 5. FESEM images of (a) CNMY1 and (b) CNMY2 obtained at optimal conditions.

#### 3.1.2 Optimization Study

Numerical optimization was applied to determine the optimal growth conditions for CH<sub>4</sub> and C<sub>2</sub>H<sub>2</sub> decomposition. Thus, the predefined criterions for the optimal working conditions were applied by keeping the synthesis parameters within the prescribed upper and lower limits of the growth conditions in order to achieve the maximum yield and the highest desirability value which will confirm the adequacy of the predicted models. Table 3 indicated that the predicted optimum conditions for CNMY1 were 950°C, 60 min, and 1.0 corresponding to growth temperature, growth time and H<sub>2</sub>/CH<sub>4</sub>, respectively leading to yield ~ 37.0 %. A yield of ~ 155 % for CNMY2 was obtained at the optimal conditions of a 560°C, 38 min and  $1.0 \text{ H}_2/\text{C}_2\text{H}_2$  (Table 4).

#### 3.2 Characterization 3.2.1 Morphology

The FESEM and TEM images of the nickel-doped activated carbon (Ni/PAC) show a successful imbedding for the catalyst particles in the pores of PAC substrate (Fig. 4a and 4b). The elemental analysis attained from EDX (Fig. 4c) shows that the loaded substrate is comprised of 88.58 wt% carbon, 7.48 wt% oxygen, wt% 3.26 wt% nickel and 0.67 wt% silicon. The growth of various types of CNMs

from this low catalyst amount validates the catalyzing role of PAC and its ability to provide good media for catalyst dispersion (Li *et al.* 2014).

Fig. 5 shows the morphologies of hierarchical nanomaterial shaped from the methane (CNMY1- $CH_4$ ) and acetylene (CNMY1- $C_2H_2$ ) decomposition at the optimal growth conditions. The hybrid CNMY1-CH4 displayed in Fig. 5a has groove-like form and resemble the PAC porous structures. CNMY1-CH4 hybrid is comprised from short interposed carbon nanotubes (CNTs) which are randomly oriented in the substrate matrix and embedded on the solid walls of the pores the activates carbon substrate. The hybrid CNMY1-C<sub>2</sub>H<sub>2</sub> in Fig. 5b and 5c, which was obtained at the optimal synthesis conditions, show bushy and graphitized tubular structures of CNTs with 10-40 nm, and an intercalated catalytic nickel nanoparticle (CNP) which was separated from the substrate and confined at the tip of the grown CNT. This observation suggests a tip growth mechanism for the CNT which triggered by the precipitation of the generated carbon fragments on the Ni surface followed by the growth of the tube and terminated finally due to the catalyst deactivation (Jeong et al. 2016).

#### 3.2.2 TGA Analysis

The TGA analysis in Fig. 7 assesses the quality and thermal stability of CNMY1 and CNMY2 obtained from CH<sub>4</sub> and C<sub>2</sub>H<sub>2</sub>. The TGA profile can be highlighted by the initial gradual loss due to moisture evaporation of 2.0 % and 5.0 % followed by constant drops to the onset combustion temperature at 507 and 455 °C for CNMY1 and CNMY2, respectively. The third region was featured by a steep drop region and ended with the oxidization section of the remaining amorphous carbon at temperature higher than 500 °C. Additionally, a well-graphitized structure for CNMY1 is obtained and confirmed by its high onset temperature. The TGA analysis indicates that the produced carbon nanostructures from both hydrocarbon precursor sustain a temperature as high as 800 °C (Zhou et al. 2014).

#### 3.2.3 BET Surface Area

The surface area of the PAC, Ni/PAC, CNMY1 and CNMY2 was determined by nitrogen adsorptiondesorption measurements. The results illustrated in Table 5 show a reduction in the surface area of catalyst impregnation. Ni/PAC due to the Furthermore, an enhancement in the surface area of the obtained hybrids CNMY1 and CNMY2 because the added structure on the substrate. The total pore volume of CNMY1 and CNMY2 reached 0.29 cm<sup>3</sup>/g and 0.44, respectively. This logical result can be ascribed to the development of new micropores resulting from the growth of different nanostructures on the substrate which will be beneficial for improving the adsorbent sorption capacity (Wang et al. 2017).

#### 3.2.4 Raman Analysis

Raman analysis displayed in Fig. 7 for the CNMY1 and CNMY2 detected the main D and G characteristic band. The D peak at around 1316 cm<sup>-1</sup> suggests the presence of disordered graphite structure while the C-C stretching vibration is characterized by the G band at around 1594 cm<sup>-1</sup>. The high intensity of D band confirms the defective nature of the obtained structure due to the thermal treatment step. Also, the grown hybrids involved multiwall structure confirmed by the disappearance of the radial breathing mode (RBM) at wavelength below 400 cm<sup>-1</sup>(Ahmad et al. 2018). The organizational degree of the obtained hybrids can be represented by  $I_D/I_G$  and it is noticed that synthesized nanocarbon structure ratio was 0.9 and 0.82 from the decomposition of  $CH_4$  and  $C_2H_2$ , respectively. This indicates that the decomposition of C<sub>2</sub>H<sub>2</sub> enhanced the growth of highly graphitized nanostructure which was also observed in the TGA analysis. Also, the CNMs synthesized from acetylene pyrolysis at750°C demonstrated I<sub>D</sub>/I<sub>G</sub> ratio of 0.74 which suggests a lesser graphitic structure than that obtained at 560°C.

#### 3.2.5 FTIR Analysis

The functional group and surface chemistry of CNMY1 and CNMY2 synthesized at the optimal growth conditions were presented in the FTIR spectroscopy in Fig. 8. Absorption bands at 3450–3500 cm<sup>-1</sup> assigned to the –OH group. The ascribed peaks from the Ni catalyst carbonyl were found at 1979 and 2138 cm<sup>-1</sup> (Chavan *et al.* 2012), whereas the peak emerged at ~ 2350 cm<sup>-1</sup> is attributed to the presence of aromatic  $sp^2$  C–H stretching vibration. The aromatic rings and C=C stretch were appeared at 1447 and 1563 cm<sup>-1</sup>, respectively. It is observed that the CNMY2 hybrids has fewer functional groups than that produced from CH<sub>4</sub> decomposition, accordingly higher hydrophobicity is expected for CNMY2 (Ezzeddine *et al.* 2016)

#### 3.2.6 Zeta Potential and Contact Angle

The adsorption mechanism is greatly affected by the surface charge and the hydrophobicity of the adsorbent (Aljumaily *et al.* 2018). Ni/PAC is not a hydrophobic material and its cast film a contact angle of  $65^{\circ}$  as presented in Table 6.

The CNMY1 has lower zeta potential than that obtained for CNMY2. The surface positive charge and basicity of CNMY1 could be due to the oxygen-free carbon sites ( $\pi$ - electron-rich regions) located in the basal planes. Furthermore, the CNMY1 hybrids show enhanced hydrophobicity associated with high contact angle of 138° but lower than that obtained for CNMY2. The super hydrophobic CNMs synthesized from acetylene decomposition demonstrated high contact angle of 160° with high value of zeta potential due to the mutual repulsion of CNMs surface charges.



Figure 6. TGA curves for CNMY1 and CNMY2.

 Table 5.
 BET results of PAC, Ni/PAC, CNMY1 and CNMY2.

Property	PAC	Ni/PAC	CNM Y1	CNM Y2
BET (m <sup>2</sup> /g)	101.1	97.2	164.6	333.83
Total pore volume (cm <sup>3</sup> /g)	0.09	0.07	0.29	0.44
Average pore diameter (°A)	34.89	21.29	96.19	65.09



Figure 7. Raman spectrum of CNMY1 and CNMY2.



Figure 8. FTIR spectrums for free MB, CNMY1 and CNMY2 after adsorption.

#### 3.2.7 Kinetics and Isotherm Studies

Two models were applied to analyze the kinetic data obtained from the batch experiments of MB adsorption on CNMY1 and CNMY2: pseudo-first order, and pseudo-second models (Fig. 9). The relevant parameters derived from the corresponding equations are provided in Table 7, imply that the adsorption process has not entirely followed the pseudo first-order rate expression for both adsorbents (Fig. 9a and 9b). In contrast, the applicability of the pseudo-second-order model better to describe the sorption kinetics of MB dye is clearly observed in Fig. 9c and 9d confirmed by the high correlation coefficient of the linear plots. This observation suggests the possibility of chemisorption adsorption mechanism through exchange or sharing electrons between the adsorbent and adsorbate (Subramani and Thinakaran 2017). For the isotherm study, the favorability of the Langmuir isotherm can be conveniently represented by the equilibrium parameter ( $R_L$ ); (0< $R_L$  <1) which is defined by equation (6).

$$\mathbf{R}_{\mathbf{L}} = \frac{1}{1 + \mathbf{K}_{\mathbf{L}} \, \mathbf{C}_{o}} \tag{6}$$

The values summarized in Table 8 and the linear plots presented in Fig. 10 agreed well with the Langmuir isotherm (Model 1) for CNMY1 and CNMY2 signifying monolayer adsorption with heterogeneous surface binding, however, sorption capacity values of CNMY2 was conveniently described by Freundlich model (Model 2) as well. This kind of duality has been stated in many publications (Wang *et al.* 2014). Comparison of the maximum adsorption uptake of MB on several adsorbents are presented in Table 9. The as-prepared hybrid carbon nanomaterials in the present work have demonstrated noteworthy adsorption uptake of 250 and 174 mg/g for CNMY1 and CNMY2, respectively.

 Table 6. Zeta potential results for Ni/PAC and the obtained carbon structures.

Sample	Zeta potential (mV)	Contact angle (°)
Ni/PAC	+1.67	65
CNMY1 (950 °C)	+9.4	138
CNMY2 (560 °C)	- 34.3	160

Model	Equation	Parameters	Valu	ies
		-	CNMY1	CNMY2
Pseudo-First-Order		-		
	$\ln(q_e - q_t) = \ln q_e - K_1 t$	$R^2$	0.83	0.89
		$K_{I}$	0.02	0.03
		$q_e$	27.0	38.9
Pseudo-Second-Order	$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t$	$R^2$	0.999	0.99
		$K_{I}$	0.002	0.0015
		$q_e$	145.0	122.7

## Table 7. Experimental values of kinetics model's constants.

## Table 8. Linearized equations of studied isotherm models.

Model	Equation	Parameters	Valu	es
			CNMY1	CNMY2
Langmuir		$q_m$	250.0	174.0
	$\frac{C_e}{C_e} = \frac{1}{C_e} + \left(\frac{1}{C_e}\right)C_e$	$K_L$	0.65	1.5
	$q_e  K_L q_m  (q_m)$	$R^2$	0.98	0.99
		$R_L$	0.03	0.01
Freundlich	1	$R^2$	0.86	0.93
	$lnq_e = lnK_f + \frac{-}{n}lnC_e$	$K_{f}$	85.0	83.7
		n	2.8	2.6

Table 9. maximum adsorption uptake (qm) comparison with previous studies.

Adsorbent	$q_m (mg/g)$	Reference
CNMY1	250	The present work
CNMY2	174	The present work
MWCNTs	109.31	(Zohre et al. 2010)
Activated carbon	123	(Suresh et al. 2011)
MWCNTs by CVD (acetylene, Fe/Si)	50.25	(Liu et al. 2014)
Cotton stalk	147.06	(Deng et al. 2011)
Graphene/magnetite	43.08	(Ai et al. 2011)
Oxidized-CNTs	99.83	(Norzilah et al. 2011)
Calcined titanate NT	133.33	(Xiong et al. 2010)
MWCNTs	59.7	(Wang et al. 2012)
Fe <sub>3</sub> O <sub>4</sub> – MWCNTs (HNO <sub>3</sub> )	48.06	(Ai et al. 2011)



Figure 9. Fittings of Pseudo-first order and Pseudo-second order kinetics models for MB adsorption on CNMY1 (a, b) and CNMY2 (c, d), respectively.



Figure 10. Langmuir and Freundlich model plots for MB adsorption on CNMY1 (a,b) and CNMY2 (c,d), respectively.

## 4. CONCLUSION

The growth of CNMs from different precursors and optimizing their growth parameters were investigated. The optimal growth conditions for the hybrid carbon nanomaterials obtained from methane and acetylene were found at the temperature of 950, 60, H2/CH4 of 1.0 and 560 °C, time of 38 min, and H2/C2H2 of 1.0, respectively. Undoubtedly, the carbon source played a significant role to affect the morphology of CNMs deposits due to discrepancies in their pyrolytic behaviors. Methane has the simplest chemical structure and comparatively stable at hightemperature than acetylene. Also, the Gibbs free energies of the C2H2 has lower decomposition temperature than methane which gives rise to lower growth temperature of CNMs. The MB removal process on each adsorbent was fitted well to a pseudosecond order kinetics model and the adsorption system was excellently presented by Langmuir isotherm model with a maximum adsorption capacity of 250 and 174 mg/g for CNM/PAC-CH4 and CNM/PAC-C2H2, respectively.

## **CONFLICT OF INTEREST**

The authors declare no conflicts of interest.

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