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Comparative Study of Chemical and Green Preparation of Mesoporous Silica

Sara K. Dawood**, Sameer H. Kareem

Department of Chemistry, College of Science for Women, University of Baghdad, Baghdad, Iraq.
*Corresponding Author.

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Abstract

The present research includes a comparative study of preparing mesoporous silica using two different methods: the chemical method and the green method. The sol-gel method was used using sodium silicate (glass water) as a cheap source of silica and the cationic-type surfactant (Hydroxylethyl, 2-Hydroxylcetyl (2-Dimethyl ammonium chloride) in the chemical method, and Zyziphus Spina Christi (Sider tree) tree leaf extract in the green method, as a soft template. The mesoporous silica prepared using the two methods were compared by characterizing them using different methods: measuring surface area by BET method and porosity by BJH, XRD, SAXRD, AFM, FTIR, SEM, and EDX. The results showed that the silica prepared by the green method had the best surface area, recording 800 m^2g^{-1} and an average pore diameter equal to 2.5 nm, while XRD measurements showed that both methods produced non-crystalline (amorphous) silica, while SAXRD showed that the green method produced more regular silica. SEM images displayed gr-SiO_2 particles were comparatively smaller than the particles of ch--SiO_2 with spherical shape particles and agglomeration as a flower shape, while EDX results confirmed the presence of Si and O, with small amount of C which may be due to non-complete decomposition of surfactants. The Langmuir, Freundlich, and Temkin isotherm models were fitted to the data obtained from the experiments and the results of adsorption study shows that the chemical prepared silica has higher capacity (63.694 mg g^{-1}) toward amlodipine adsorption according to Freundlich parameters obtained by linear fitting.

Keywords: amlodipine, cationic surfactant, mesoporous silica, soft template, Zyziphus Spina leaves Christi.

Introduction

Mesoporous silica (SiO_2) is widely used in several applications, such as adsorption and catalysts technology, building industry, agriculture, food industry, and medical formulations, because of their preparation simplicity, stability, non-toxicity, biocompatibility, and to controllable particle sizes[1-3]. Different types of mesoporous silica, which are different in terms of pore size, shapes, symmetry and geometry, were synthesized by varying the starting materials, the template, and the reaction parameters, e.g. Mobil Crystalline of Materials (MCM)[4, 5], Santa Barbara Amorphous (SBA)[6], Hollow Mesoporous Silica (HMS)[7], and Technische Universiteit Delft (TUD)[8].

In the mesoporous silica synthesis, substantial quantities of chemical surfactants as soft templates have been used. These surfactants and their by-products through template removal, had an adverse effect on the ecosystem, such as threat to aquatic life[9, 10], in addition, the process wastes are costly organic templates that require a significant amount of time and effort[11]. So, some researchers used biodegradable templates. Non-ionic surfactants of sorbitan ester, which are commonly utilized in

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cosmetics, food, or pharmaceutical products, influence the pore diameter with an average of 3 nm[12]. As ecofriendly methods, natural polysaccharides (such as starch) which are obtained from variety of things including grains and vegetables were used as a green template to create nanostructured mesoporous silica[13]. Wormhole-like pore mesoporous silica was synthesized by a green method using Tween-20 and starch, which are FDA-approved surfactants, in place of pricy and dangerous surfactants like CTAB[14]. A natural biopolymer lignin extracted from coir pith was used in the synthesis of silica nanoparticles as a soft template, capping, and stabilizing agent and characterized by different techniques[15].

Zizyphus Spina Christi (ZSC) is a tree present in south and middle Iraq and some Middle Eastern nations like Jordan, Egypt, and Iran. The leaves extract contains a naturally occurring, biodegradable surfactant saponin in addition to flavonoids and other components[16, 17]. Pharmaceutical industry has become one of the main causes of the pollution of aquatic ecosystems because of the unused medication that is being disposed of. The majority of these compounds seriously harm aquatic life.

One medication that meets the criteria for being an emergent pollutant is amlodipine besylate, which is commonly used for the treatment of high blood pressure[18, 19].

According to our knowledge, we believe that the use of Sidr tree leaf extract as a biodegradable and environmentally friendly surfactant as a soft template in the preparation of mesoporous silica is new, as well as the use of the resulting silica in studying its adsorption capacity for the drug amlodipine. So, the aims of this study are to prepare mesoporous silica from inexpensive silica source using natural biodegradable surfactant as soft template, and to study their properties in comparison with chemically prepared silica using some precision analysis techniques such as such as XRD, N_2 adsorption desorption analysis, AFM, SEM, and EDX. In addition, adsorptive ability was also comparing between them.

Materials

Hydroxyl ethyl, 2- Hydroxyl cetyl (2-Dimethyl ammonium chloride), Dehyquart (E-CA) (28%, its color white yellow, chemical formula is HO-$(CH)_2$ $N(Me)_2CH_2CH(OH)$ $(CH_2)_{13}Me$, M. Wt. = 366 g/mol) and sodium silicate (glass water)(14%NaOH, 27%SiO_2) were bought from a government-owned plant oil - Baghdad- Iraq. The leaves of Zyziphus Spina Christi were provided from Baghdad – Iraq.

XRD and SAXRD, Siemens model D500, Cu Kα (λ =1 .541Å), was used to discover the crystallinity and the order of mesopores of the prepared silica, and the evaluation of the diffractograms took place between 2Θ = 10–80 and 2Θ = 1–10, respectively. The adsorption desorption analyzer [Type: NOVA 2200e-Quantachrome Analyzer], was used to evaluate the BET surface area and BJH porosity. The morphology of silica was characterized by SEM (Oxford instruments model SEM: S-3200N).

Methods

Green surfactant was extracted from the leaves of Zyziphus Spina Christi as follows: The leaves were washed with tap water and then with distilled water to get rid of accumulated dirt and other contaminants. The cleaned leaves were ground into a fine powder after being sun-dried. 100 g of the fine powder with 300 mL of mixture of distilled water and ethanol (50% v/v) was heated overnight at hot plate and the temperature was set at 60˚C, after that the mixture was filtered and the supernatant was stored for further usage.

To prepare mesoporous silica by chemical method, the following procedure was employed which is comparable to the technique we previously reported[20]: The dilute sodium silicate (150 mL) was added dropwise to the heated solution at 80 °C of 3.5 g of Dehyquart (150 mL, mixture of 50% water and ethanol v/v) through 3hrs. After aging for 24 hrs, the precipitate was collected, dried for six hrs at 50 °C, and then calcined for four hrs at 600 °C. For green method, the same procedure was employed but the heated solution was 150 mL of the extracted solution of Zyziphus Spina Christi leaves.

Results and discussion

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Characterization

FTIR Analysis

FTIR spectrum in Fig. 1 shows some bands due to the presence of functional groups which confirm the formation of SiO_2. The silanol group's bending and stretching bands were observed at 3435.22 cm^{-1} and 1635.64 cm^{-1} respectively, while the bands at 1085.92 cm^{-1} and band at 800.46 cm^{-1} are associated with asymmetric and symmetric stretching of the Si–O–Si bond respectively, the band identified at 468.70 cm^{-1} is due to Si–O–Si bending[21- 23]. The stretching bands at 2920.23 cm^{-1} and 2850.79 cm^{-1} and the bending at 1477.47 cm^{-1} appear in Fig. 1 are assigned to stretching and bending of C-H bond respectively, which due to presenting the organic surfactant. Disappearance of these peaks in Fig. 1 confirms the decomposition of surfactant and the purity of obtained silica.

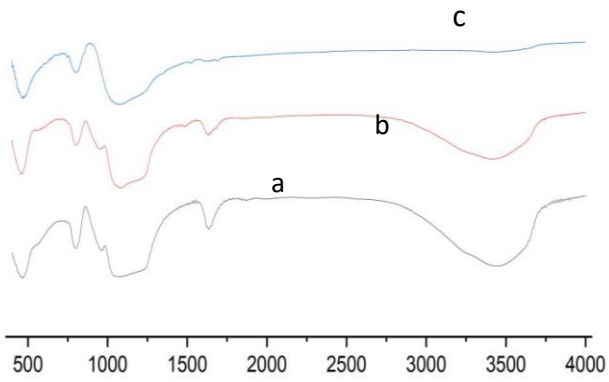

Figure 1. FTIR spectrum of a: ch-SiO_2 before calcination, b: gr-SiO_2 before calcination, c: SiO_2 after calcination.

XRD Analysis

As Fig. 2 shows, wide and small angle XRD displayed patterns corresponding to mesoporous silica. The broad peak at the range 2Θ = 20 to 2Θ = 30° found in wide angle XRD is particular to silica that is amorphous, and it matched well with JCPDS Card No. 20-1050[24]. The low angle XRD pattern Fig. 3 for chemically prepared silica shows a broad, strong peak with a shoulder at about 2Θ = 1.85°, while for green prepared silica the peak appears at 2Θ = 0.9°, displaying a connected framework mesopore distribution. The peaks at 2Θ= 3.2, and 2.3° for chemically and green prepared silica respectively, due to regular pore structure[25, 26]. The data is consistent with other studies[27].

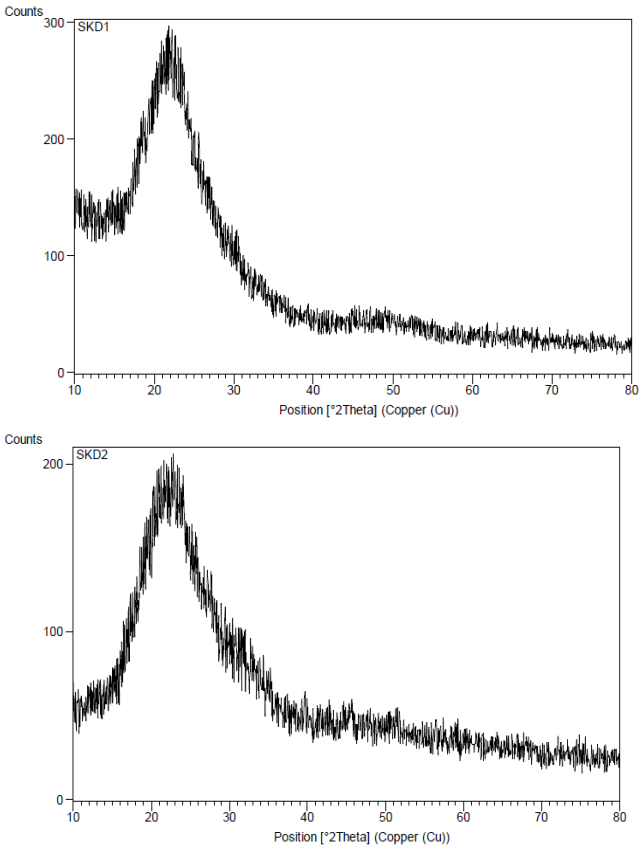

Figure 2. Wide-angle XRD patterns of a: ch-SiO_2, b: gr- SiO_2.

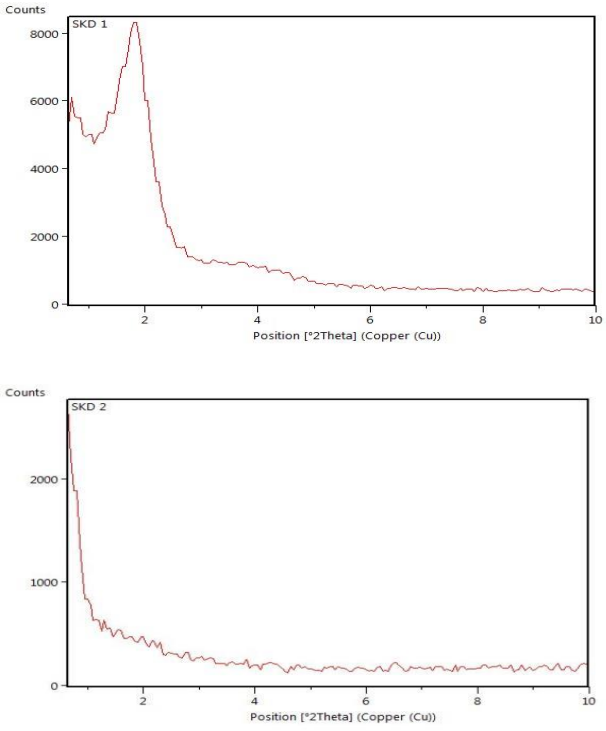

Figure 3. Small-angle XRD patterns of a: ch-SiO_2, b: gr- SiO_2.

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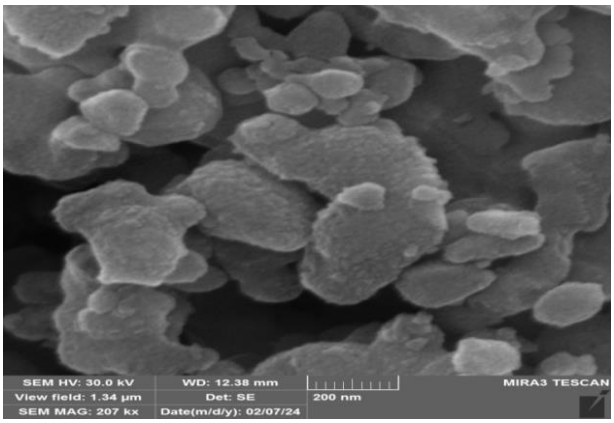
SEM/EDX Analysis

The SEM/EDX analysis of ch-SiO_2 and gr-SiO_2 were carried out for the two samples' morphology and compositions. Figs. 4 and 5 display the SEM images which showed that the gr-SiO_2 particles are comparatively smaller than the particles of ch-SiO_2 with spherical shape particles and agglomeration as a flower shape. To ascertain the elemental composition, EDX analysis was performed and the results confirmed the presence of Si and O, with small amount of C which may be due to non-complete decomposition of surfactants.



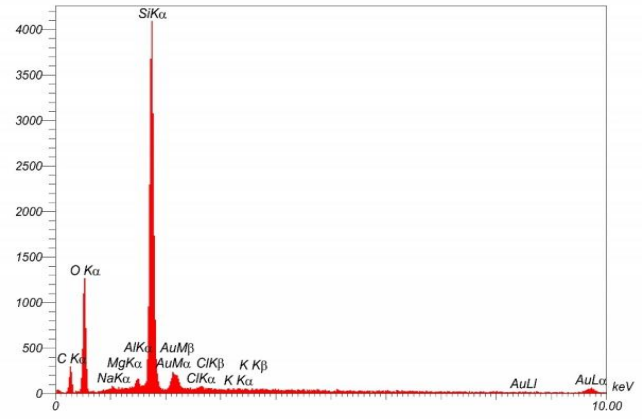
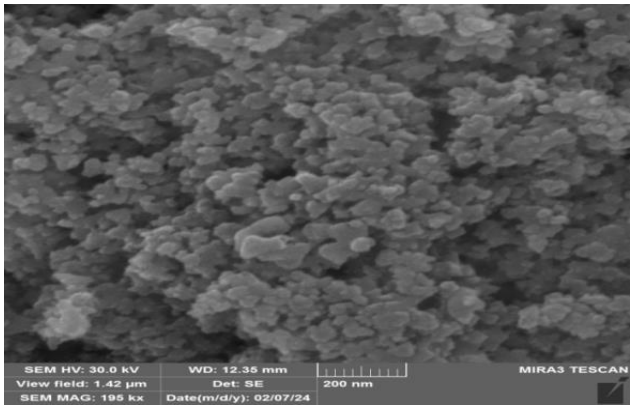


Figure 4. SEM pictures and EDX spectrum of ch-SiO_2.



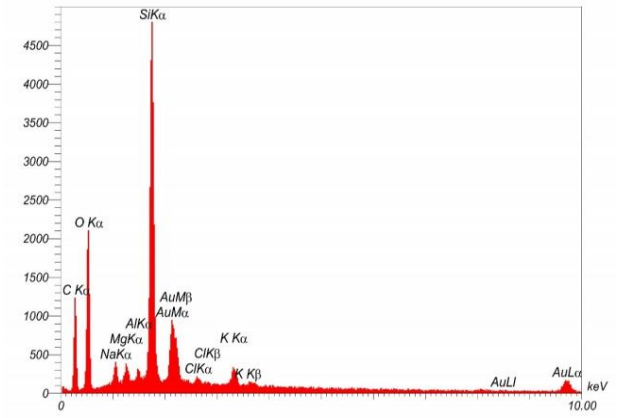
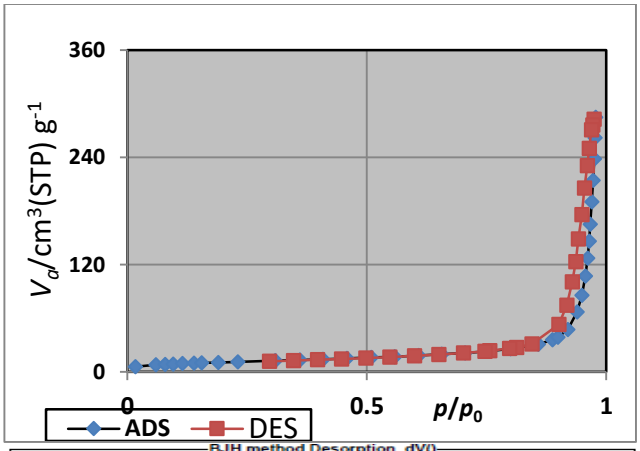


Figure 5. SEM pictures and EDX spectrum of gr-SiO_2.

Surface Area and Porosity

Surface area and porosity determination of the two mesoporous silica particles are illustrated in Figs. 6 and 7 which show the plots of adsorption-desorption isotherm and pore size distribution, and the parameters, as shown in the Table 1, were obtained from the BET and BJH methods.



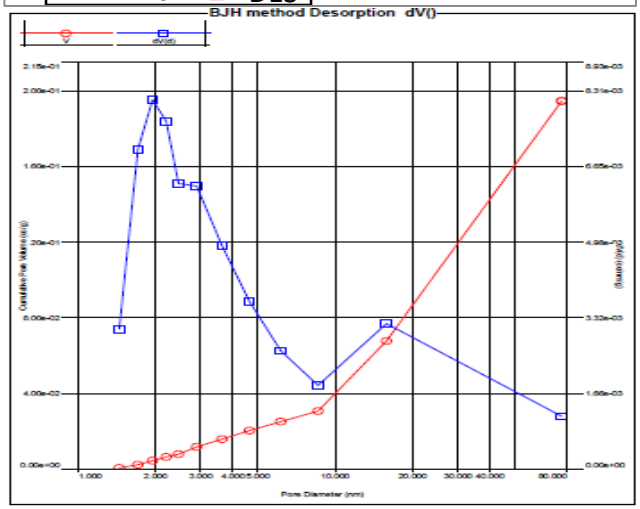


Figure 6. a: N_2 adsorption desorption isotherm, b: distribution of the pore size of ch-MPS.

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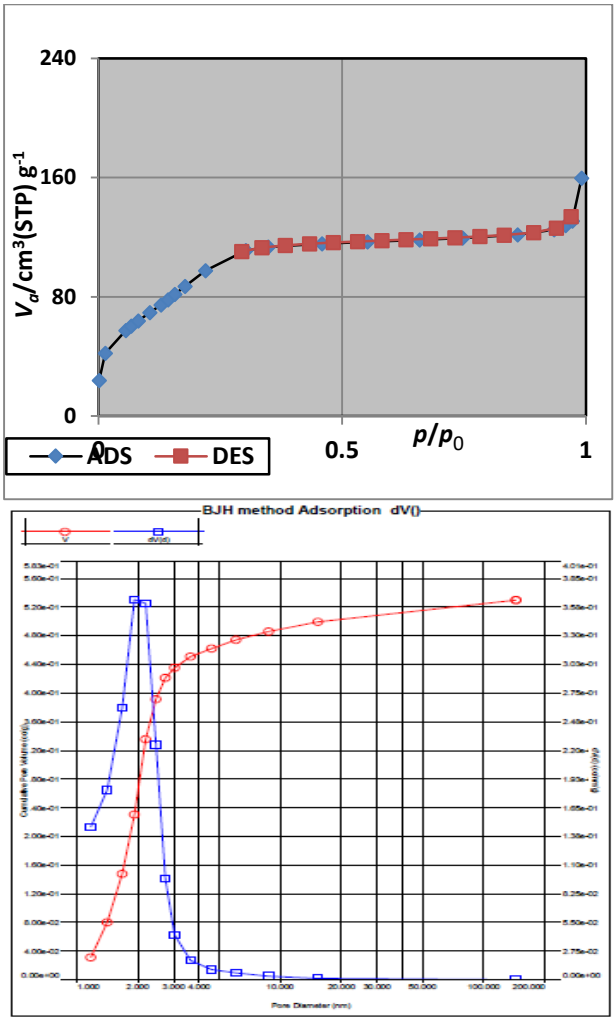


Figure 7. a: N_2 adsorption desorption isotherm, b: distribution of the pore size of gr-SiO2.

The acquired isotherms in Figs. 6a and 7a for ch-SiO_2 and gr-SiO_2 are typical type –IVa and IVb isotherms respectively, where IVa exhibits an H1 type hysteresis loop and IVb without hysteresis loop indicating the production of mesoporous with cylindrical shape pores. Type IVa isotherm accompanied by hysteresis which occurred when the pore is wider than ~ 4 nm[28, 29], however, fully reversible Type IVb isotherms are seen in adsorbents with narrower mesopore widths. Additionally, conical and cylindrical mesopores with tapered ends closed provided this isotherm (Type IVb) [30].

Figs. 6b and 7b for pore size distribution(PZD) show that the values of PZD for gr-SiO_2 has a narrow range of pore size distribution and displayed the range 2-4 nm, while the PZD of ch-SiO_2 has wide range and displayed the range 2-20 nm. The results of isotherms and PZD also showed the presence of mesoporous in the two samples.

Table 1. Texture properties of the two adsorbents.

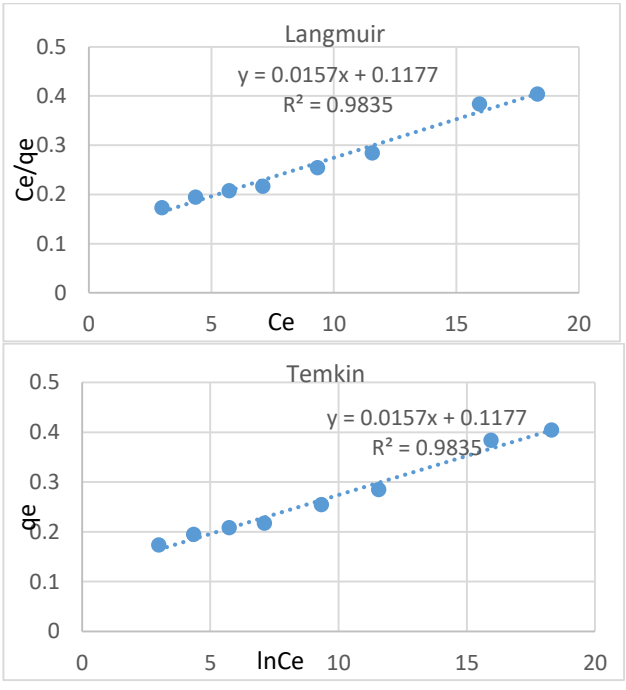
Silica	Surface area m^2g^{-1}	Pore diameter nm	Pore volume cm^3g^{-1}
ch-SiO_2	47.5	16.4	0.195
gr-SiO_2	817. 7	2.25	0.529

The results of Table 1. demonstrate that the gr-SiO_2 has surface area higher than ch-SiO_2 which are 817. 7 and 47.5 m^2g-^{1}, has smaller mean pore diameters which are 2.25 and 16.4 nm (in the mesoporous range), and has higher pore volume which are 0.529 and 0.195 cm^3g^{-1} respectively.

Adsorption Modeling

ch-SiO_2 and gr-SiO_2 are used as adsorbents to perform the adsorption process for one adsorbate which is amlodipine drug. The adsorption studies were conducted employing the batching method. To this end, 70mg of absorbent, 100 mL adsorbate solution, 115 min equilibrium time, and at temperature 303 K were applied to perform the adsorption experiments.

The experiments data were analyzed by three isothermal models: Freundlich, Langmuir and Temkin, to evaluate the effectiveness of adsorption. Figs. 8 and 9 show the linear plots of the experimental data for the adsorption of amlodipine onto the two type of mesoporous silica, while Table 2. summarizes the obtained parameters of the three models.



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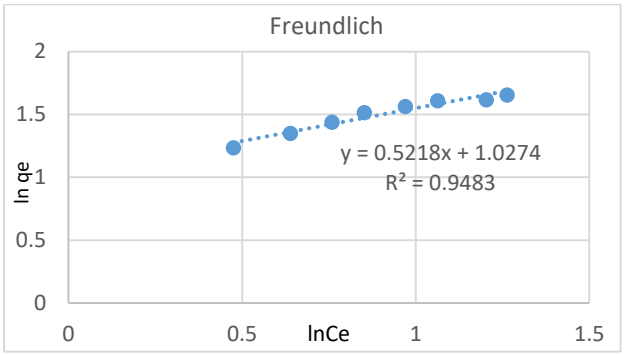
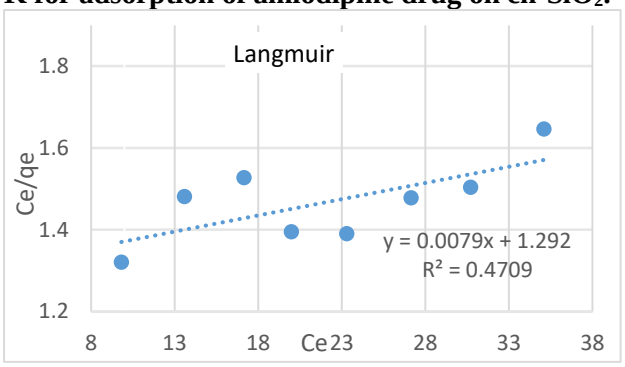
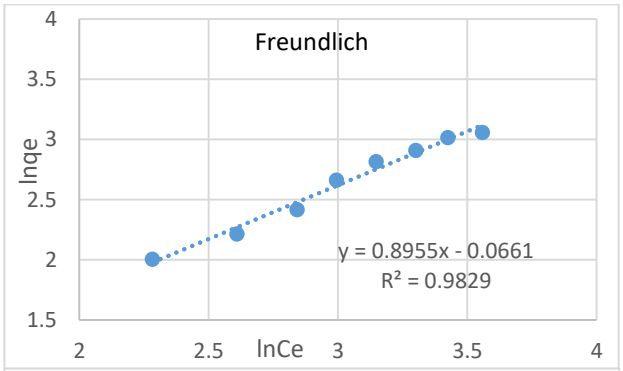


Figure 8. Linear plots for the three models at 303 K for adsorption of amlodipine drug on ch-SiO_2.





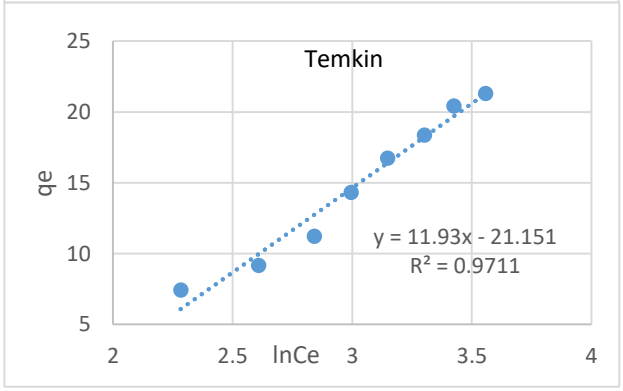


Figure 9. Linear plots for the three models at 303 K for adsorption of amlodipine drug on gr-SiO_2.

Table 2. The three isotherm parameters gained by linear fitting of amlodipine adsorption on the two surfaces.

Sample	Langmuir			Freundlish			Temkin		
	R^2	K_L	Q_m (mgg^{-1})	R^2	K_F	n	R^2	lnK	B $Jmol^{-1}$
ch-SiO_2	0.984	0.134	63.694	0.957	0.378	1.916	0.985	0.007	0.063
gr-SiO_2	0.471	0.006	126.58	0.956	0.243	1.117	0.9561	-0.823	0.937

As stated by the correlation coefficient (R^2) values, the outcomes of the experiment follow the three models but the Langmuir and Temkin models better than Freundlich isotherm. Based on the information, one can deduce that the amlodipine drug form a monolayer on homogenous ch-SiO_2 surface, and the maximum adsorption capacity was 63.694 mg/g. For gr-SiO_2 surface, Freundlich and Temkin isotherms are suitable to estimate amlodipine adsorption, but Langmuir isotherm does not adequately explain how the adsorption mechanism works according to R^2 values[18, 31, 32]. So the comparison was made through the Freundlich isotherm parameters, K_F and n, which demonstrates that the surface of ch-SiO_2 has higher capacity than gr-SiO_2, and the amlodipine adsorption favorable multilayer adsorption process on gr-SiO_2 surface, while on ch-SiO_2 surface it is form a monolayer[33, 34].

Conclusion

From the characterization results and experimental data of the study of chemical and green preparation of mesoporous silica and their adsorptive capability toward amlodipine drug, the following can be concluded:

The study demonstrated that two types of mesoporous silica could be synthesized using chemical and biodegradable surfactants, and the characterization techniques indicate that the gr-SiO_2 mesoporous silica particles possess smaller pore diameters and a larger surface area than mesoporous silica particles of ch-SiO_2. This indicates that this silica can be used in different applications especially in adsorption processes. The experimental

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adsorption data of the two mesoporous silica shows a respectable degree of agreement with the Freundlich equilibrium model, as evidenced by the high R^2 values (>0.95). This reveals that the surfaces of the two prepared mesoporous silica are heterogonous. The adsorption capacity of ch-SiO_2 surface was 63.694 mg g^{-1} for amlodipine drug which was higher than adsorption capacity of gr-SiO_2. This reveals that the synthesized ch-SiO_2 was effective adsorbent for the removal of amlodipine from water.

Acknowledgment

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Author's Declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for re-publication, which is attached to the manuscript.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- No potentially identified images or data are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Baghdad.

Authors' Contribution Statement

S. H. K. suggested and planned the research idea. S.K.D. designed and carried out the experiments. Both authors contributed in the analysis and discussing of the results and writing the manuscript.

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دراسة مقارنة للتحضير الكيميائي والخضراء للسيليكا ذات المسام المتوسطة

ساره قحطان داود، سمير حكيم كريم

قسم الكيمياء، كلية العلوم للبنات، جامعة بغداد، بغداد، العراق

الخلاصة

يتضمن البحث الحالي دراسة مقارنة لتحضير السيليكا مسامية باستخدام طريقتين مختلفتين: الطريقة الكيميائية والطريقة الخضراء. تم استخدام طريقة gel-sol باستخدام سيليكات الصوديوم (الماء الزجاجي) كمصدر رخيص للسيليكا والمواد الخافضة للتوتر السطحي الكاتيونية في الطريقة الكيميائية، ومستخلص أوراق شجرة السدر بالطريقة الخضراء كقالب ناعم، وتمت مقارنة السيليكا المتوسطة المسام المحضرة باستخدام الطريقتين من خلال توصيفها باستخدام طرق مختلفة: قياس مساحة السطح بطريقة BET والمسامية بواسطة BJH، XRD، SAXRD، AFM، SEM و EDX أظهرت النتائج أن السيليكا المحضرة بالطريقة الخضراء حصلت على أفضل مساحة سطحية مسجلة 800 م^{2}جرام$^{-1}$ ومتوسط قطر مسام يساوي 2.5 نانومتر، بينما أظهرت قياسات حيود الأشعة السينية أن كلا الطريقتين أنتجتا سيليكا غير بلورية (غيرمتبلورة)، بينما أظهرت طريقة SAXRD أن الطريقة الخضراء أنتجت سيليكا أكثر انتظاما، وأظهرت دراسة الامتزاز أن السيليكا المحضرة كيميائيا لديها قدرة أعلى على امتزاز الأمليوديين وفقا لمعايير فروندليتش التي تم الحصول عليها عن طريق التركيب الخطي.

الكلمات المفتاحية: أمليوديين، خافض للتوتر السطحي الكاتيوني، سيليكا مسامية، قالب ناعم، اوراق نبات السدر.