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Preparing Mesoporous Alumina as a Delivery System for the Ciprofloxacin Drug using the Microemulsion Method

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Abstract

Two samples of γ–mesoporous alumina (0.3M/m-Al_2O_3 and 0.5M/m-Al_2O_3) were prepared using the microemulsion method, with aluminium sulphate serving as the alumina precursor. The raw materials for microemulsion are sodium dodecyl sulphate (SDS) as the surfactant, 1-butanol as a cosurfactant, and n-hexanol as the oil phase. Scanning Electron Microscopy (SEM), BET surface area, BJH porosity of the samples, and their N2 adsorption-desorption isotherms at 77 K. The X-ray diffraction (XRD) and AFM techniques were used to characterize these samples. The results show that the two samples have identical phase structures and perfect indexing to the γ-Al_2O_3; the 0.3M/m-Al_2O_3 sample has a larger surface area, 229.18 m^2 g^{-1}, pore diameter, 5.59 nm, and pore volume, 0.32 $cm^3.g^{-1}$, than the 0.5M/m-Al^2O^3 sample. The morphology of the two samples was small spherical particles aggregated in spherical agglomerates, but the 0.3M/m-Al_2O_3 sample displayed smaller particles than the 0.5M/m-Al_2O_3 sample; the percentage of aluminium oxide was high, equal to 95.5% and 97.3% by weight for the 0.3M/m-Al_2O_3 and 0.5M/m-Al_2O_3, respectively. As a model, the 0.3M/m-Al_2O_3 sample was used as a carrier for delivering the ciprofloxacin drug. The loading was performed using the impregnation method, while the release was achieved through a dialysis bag with buffer solutions at pH levels of 7.4 and 5.4. The results indicate that the sample can serve as a suitable carrier for the ciprofloxacin drug.

Article Info.

Keywords:
Microemulsion, Mesoporous Alumina, Ciprofloxacin, Drug Delivery, Release Profile.

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1. Introduction

Microemulsion is defined by The International Union of Pure and Applied Chemistry (IUPAC) as thermodynamically stable isotropically clear dispersion of two immiscible liquids, such as oil and water, stabilized by an interfacial film of surfactant molecules from 1 to 100 nm, commonly 10 to 50 nm [1-3]. Surfactant pores with diameters less than 2 nm are micropores, those with diameters between 2 and 50 nm are mesopores, those with diameters greater than 50 nm are collectively known as macropores, and those with diameters greater than 100 µm are ultramacropores. γ-Mesoporous Alumina (m-Al_2O_3) has unique characteristics, including high surface area and pore volume, hard mesopore structure, and changeable pore diameter and particle size [4,5], allowing it to be used in a wide range of applications, such as adsorption, catalyst, photo-therapy and drug delivery [6-8]. Many synthetic methods have been used to synthesize mesoporous alumina, such as ball milling [9], laser ablation [10], sol-gel [11], pyrolysis [12], hydrothermal [13], microemulsion [14], hard template [15] and soft template methods [16]. Alumina has many different crystal structures, such as δ, η, γ, θ and α phases, etc. Among them, γ and α alumina have been the center of attention. Ghosh and Naskar synthesized mesoporous γ-Al_2O_3 nanorods by a microemulsion (water in oil) method using an aqueous based alumina sol, anionic surfactant dioctyl sodium sulfosuccinate (AOT, sodium bis(2-ethylhexyl sulfosuccinate)), the non-ionic co-surfactant Span 80 (sorbitan monooleate, 1,4-Anhydro-D-glucitol 6-[(9Z)-octadec-9-enoate) and cyclohexane as an oil phase [14].

Chang et al. prepared m-Al_2O_3 spheres by the microemulsion method using aluminium isopropoxide as the raw material and water/dimethyl benzene emulsion as reaction media [17]. Zhang et al. prepared large mesoporous alumina by the hydrolysis of aluminum tri-sec-butoxide in the presence of cetyltrimethylammonium bromide using oil-in-water microemulsion template [18]. Shiraz et al. prepared three large mA powders by the microemulsion method with different microemulsion structures and compositions (various surfactants and oil phases) and employed them as catalyst carriers for the preparation of mesoporous nanocrystalline Ni/Al_2O_3 catalysts [19].

Mesoporous alumina's application in drug delivery is becoming more and more focused on its potential as carriers of both small and large molecules, because drug-loaded m-Al_2O_3 demonstrated a long-term release profile, which is the best possible system for delivering drugs [20]. In our laboratory, different carriers of mesoporous silica were prepared for different delivering drugs such as ciprofloxacin [21, 22], metoprolol [23] amlodipine [24] and investigated.

The aim of the present research is to prepare m-Al_2O_3 as a carrier for delivering ciprofloxacin drug using the microemulsion method. The raw material for alumina was aluminium sulfate while for microemulsion were sodium dodecyl sulfate as surfactant, 1-butanol as co-surfactant and n-hexanol as an oil phase. The loading was performed by the impregnation method, while the release was by the dialysis bag with buffer solution of pH 7.4 and 5.4.

2. Experimental

2. 1. Chemicals

Aluminium sulfate ($Al_2(SO_4)_3$) as alumina precursor was obtained from Panreac Química (99%), sodium dodecyl sulfate (SDS, $CH_3(CH_2)_{11}OSO_3Na$,98%) as surfactant was obtained from the state company of vegetable oils – Iraq, 1-butanol from BDH Limited Poole England (98%) as co-surfactant and n-hexanol as an oil phase were provided from BDH Limited Poole England (98%), and ammonium hydroxide (25%) from ACI Labscan was used as precipitation agent. The antibiotic ciprofloxacin drug, as the delivery model, molar mass 331.346 g/mol, and λ_{max} = 276 nm in aqueous solution, was purchased from SDI Company with a purity of 98%.

2. 2. Characterization

Autosorb-1 Quantachrome Instrument (Quantachrome nova, 2200E, Germany) was used to determine BET (Brunauer, Emmett and Teller) surface area and BJH (Barret-Joyner-Halenda) porosity of the samples and their N_2 adsorption–desorption isotherms at 77 K. The degassing was performed for 2 hours at 120 °C preheating temperature and at vacuum. The X-ray diffraction (XRD) patterns were obtained with a Rigaku diffractometer (Siemens model D500) using Cu Kα (λ = 0.154 nm) X-ray. In addition, Atomic Force Microscope (AFM; Afm DME Denmark) and Scanning Electron Microscopy (SEM; ZEISS model: Sigma VP) were also used for the characterization.

2. 3. The Preparation

m-Al_2O_3 as carriers were prepared using two different concentrations of aluminum sulfate (0.3 and 0.5 M/m-Al_2O_3) as aluminum oxide precursors. The procedure was as follows: 1.85 g (0.3 M) or 3.08 g (0.5 M) aluminium sulfate was dissolved in 18 mL deionized water, and 15 g of Sodium Dodecyl Sulfate (SDS) was added with stirring at 70 °C. To this mixture, 58 g of n-hexanol and 10 g 1-butanol were added with stirring for 30 minutes at 70 °C. After that, 10 mL of ammonium hydroxide

(25 %, V/V) was added with stirring for 2 hours at 70 ℃. The product was aged in a water bath for 20 hours at 70 ℃. This was followed by filtration and washing with ethanol first and deionized water secondly until the pH became 7, and the mixture was dried and calcinated at 550 ℃ [25]. The preparation procedure can be illustrated with Fig. 1.

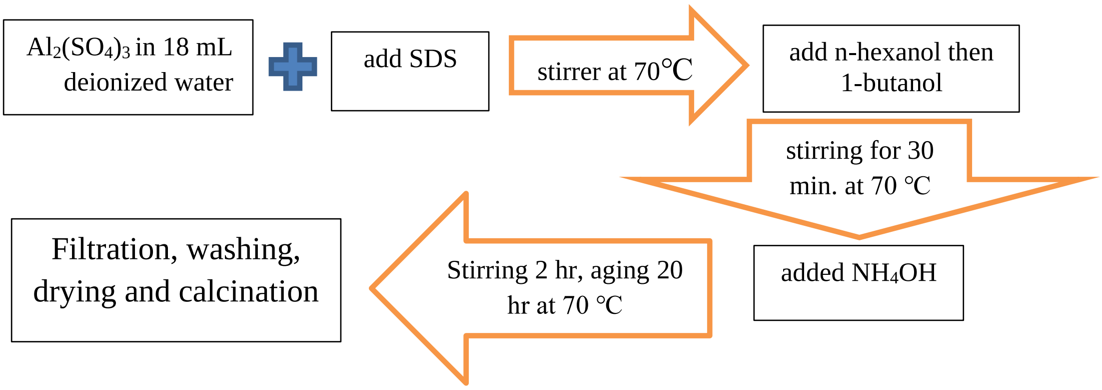

Figure 1: Schematic of the preparation procedure.

2.4. Loading and Release Experiments

The medicinal product (ciprofloxacin, CIP) was loaded on the m-Al_2O_3 carriers via impregnation as follows: 15 mL of a 30 mg.L^{-1} solution of CIP drug was added to the 0.3 g m-Al_2O_3 carrier, and then placed on a magnetic stirrer at 25 ℃ for 24 hours to achieve equilibrium. The resulting solution was filtered, and the resulting liquid was removed into a quartz cell of the UV-Vis spectrophotometer to determine the amount of ciprofloxacin. After that, the drug-loaded mesoporous composite was oven-dried for 12 hours at 50°C.

In vitro release experiment was carried out according to the following [26]: a dialysis bag containing the drug-loaded mesoporous composite with 2mL of buffer saline solution of pH 7.4 or 5.4, was soaked in the 18 mL of the saline solution and maintained at 37 ˚C under constant magnetic stirring (150 rpm). One milliliter sample were extracted at prearranged intervals (substituted by fresh Phosphate Buffered Saline, PBS). The drug concentration in the liquid phase was evaluated by UV-Vis spectrophotometery.

3. Results and Discussion

3. 1. Characterization

The two samples of the prepared m-Al_2O_3 were analyzed by XRD and their patterns are shown in Fig. 2. The depicted XRD patterns for 0.3M/m-Al_2O_3 (Fig. 2a) displays distinct peak patterns of diffraction at 20.1°, 37.17°, 39.74°, 45.99°,61.20°, 66.94° while for 0.5M/m-Al_2O_3 (Fig. 2b) the peaks of diffraction were at 20.1°,37.59°, 39.74°, 46.16°, 59.93°, 66.97° which belong to the (111), (311), (222), (400), (511) and (440) cubic planes, respectively. These two samples' results demonstrated identical phase structures and perfect indexing of all diffraction peaks to the γ-Al_2O_3 (JCPDS card No. 0.0425) [27-29].

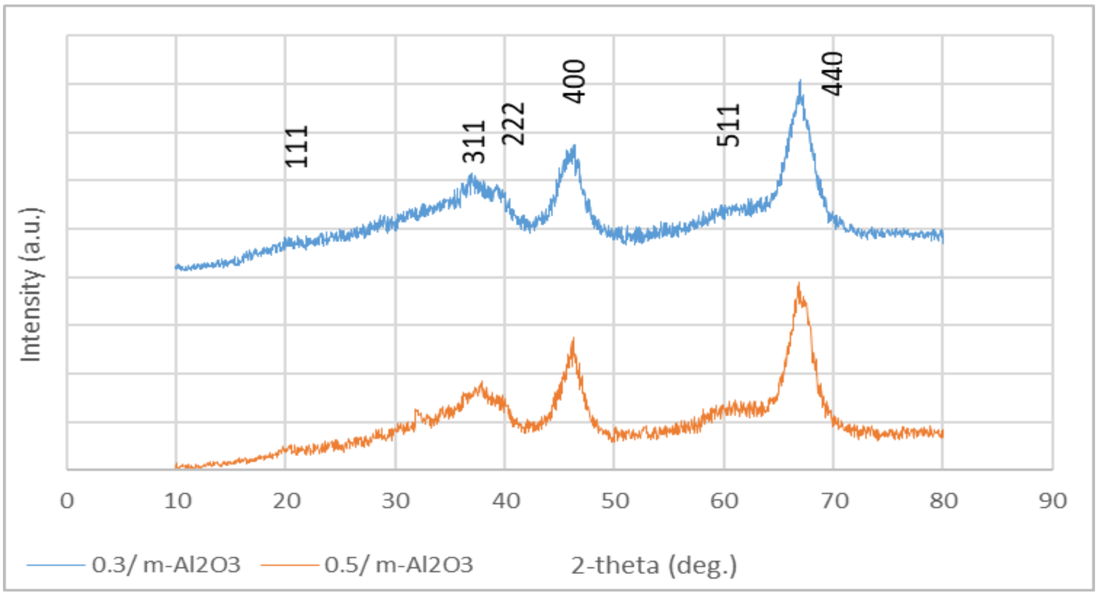

Figure 2: XRD patterns of 0.3M/m-Al_2O_3, and 0.5M/m-Al_2O_3.

Scherrer formula (Eq. (1)) [18] was used to determine the average crystallite size of these two samples using the (440) peaks; the obtained results were 5.06 and 6.32 nm for the 0.3M/m-Al_2O_3 and 0.5M/m-Al_2O_3, respectively.

$$D = \frac{k\lambda}{\beta \cos\theta} \qquad (1)$$

where D is the crystallite size, β is the peak width at half maximum intensity, k is a constant (0.94), λ is the X-ray wavelength (0.15418 nm), and Ɵ is the peak position. Figs. 3 and 4 depict the samples' N_2 isotherms and the corresponding pore size distribution.

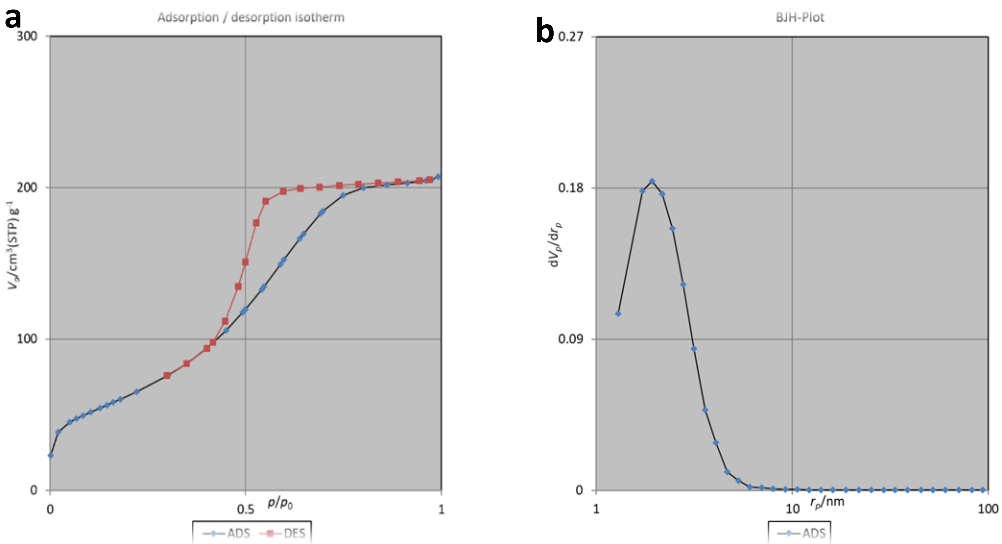

Figure 3:(a) N_2 adsorption/desorption isotherms 77K and (b) pore size distributions of 0.3M/m-Al_2O_3.

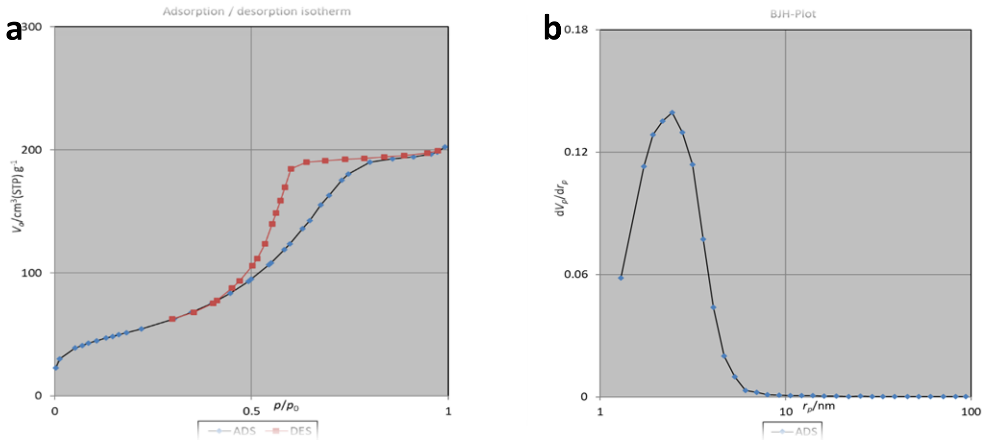

Figure 4: (a) N_2 adsorption/desorption isotherms at 77K and (b) pore size distributions of 0.5M/m-Al_2O_3.

The observed nitrogen isotherms exhibited a significant hysteresis at relative pressures P/P^0 less than 0.5 for the two samples. These isotherms were classified as type IV (a), which indicates mesoporosity. In this case, capillary condensation was accompanied by hysteresis, which occurred for pores wider than ~ 4 nm [30, 31]. The other type of hysteresis loop is H_2 (b) associated with ink–bottle pore blocking. However, the size distribution of the neck widths was significantly bigger [32]. The obtained structural properties of mesoporous alumina were: for the 0.3M/m-Al_2O_3 with a surface area of 229.18 m^2g^{-1}, a pore volume of 0.3203 cm^3g^{-1} and an average pore diameter of 5.5902 nm. For the 0.5M/m-Al_2O_3: surface area of 191.12 m^2g^{-1}, pore volume of 0.3124 cm^3g^{-1} and average pore diameter of 6.5385 nm. For comparison, the 0.3M/m-Al_2O_3 sample had higher surface area, pore diameter and pore volume than the 0.5M/m-Al_2O_3 sample. These results mean that the concentration of aluminum sulfate has a suitable effect on the surface properties of the prepared alumina, as at low concentrations the reaction is slower, which allows for the formation of pores, which is reflected in the increase of the surface area. BJH estimations for the pore size distribution based on adsorption information, as shown in Fig.3, show a small range distribution for both samples.

Fig. 5 shows the SEM analysis demonstrating that the morphology of alumina samples varied with the aluminum sulfate concentration (0.3 and 0.5 M). The two samples showed small spherical particles aggregated in spherical agglomerates. For comparison, the 0.3M/m-Al_2O_3 sample displayed smaller particles than the 0.5M/m-Al_2O_3 sample. These results agree with our results of the XRD analysis.

EDX analysis was also conducted for the two samples (Fig. 6), and the results are listed in Table 1. The percentage of aluminum oxide was high, equal to 95.5 % and 97.3 % by weight for the 0.3M/m-Al_2O_3 and 0.5M/m-Al_2O_3, respectively, with a small percentage of carbon, which may be due to incomplete burnout of surfactants and impurity from precursor [33].

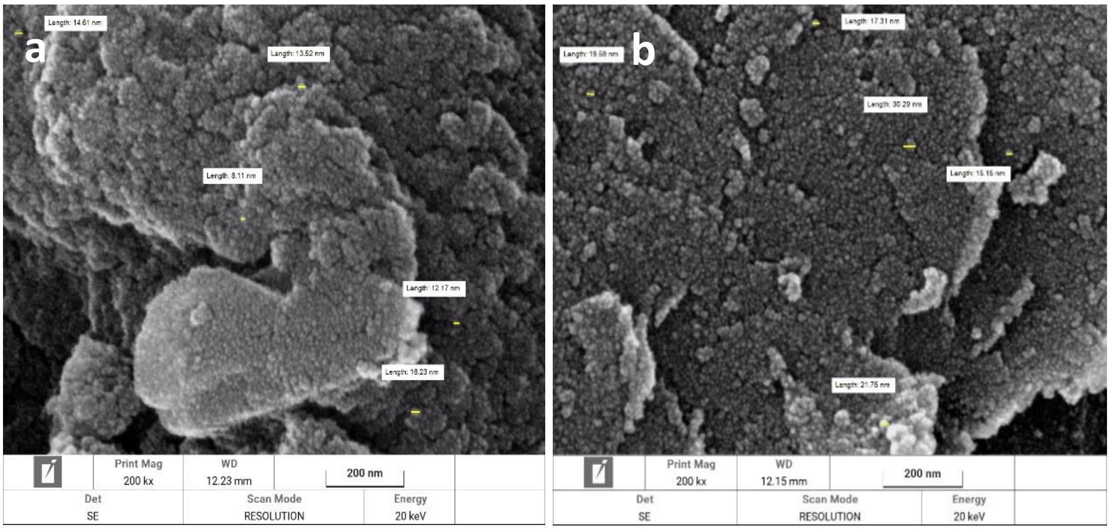

Figure 5: SEM images of (a) 0.3M/m-Al_2O_3 and (b) 0.5M/m-Al_2O_3.

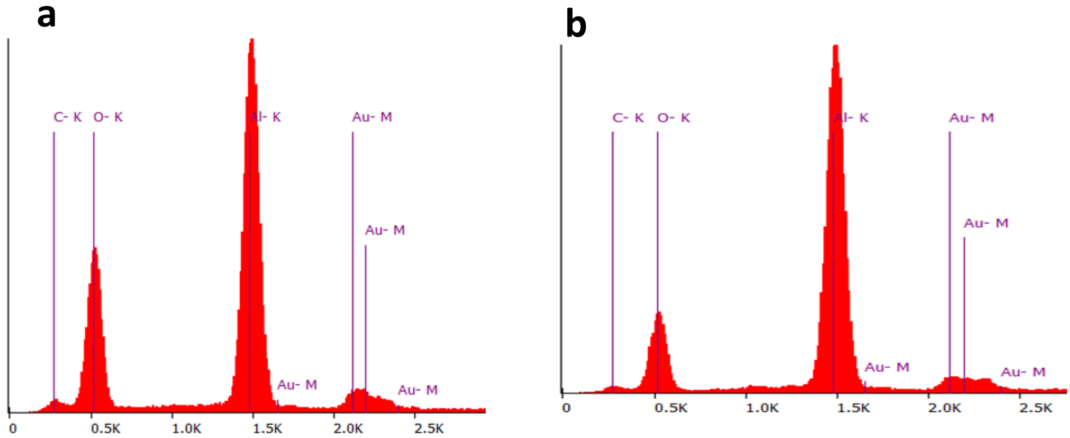

Figure 6: (a) EDX analysis for 0.3M/m-Al_2O_3 and (b) EDX analysis for 0.5M/m-Al_2O_3

Table 1: The chemical composition of 0.3M/m-Al_2O_3 and 0.5M/m-Al_2O_3.

Type	Element	Atomic%	Atomic Error%	Weight%	Weight Error%
0.3/m-Al_2O_3	C	4.2	0.6	2.5	0.4
	O	58.2	0.8	46.5	0.6
	Al	36.4	0.2	49.0	0.3
0.5/m-Al_2O_3	C	2.1	0.3	1.3	0.2
	O	61.3	0.5	49.1	0.4
	Al	35.6	0.2	48.2	0.2

Fig. 7 shows the AFM images demonstrating the two samples had small spherical particles aggregated in spherical agglomerates. For comparison, the 0.3M/m-Al_2O_3 sample displayed smaller particles than the 0.5M/m-Al_2O_3 sample, which agrees with our results of the SEM image and XRD analysis.

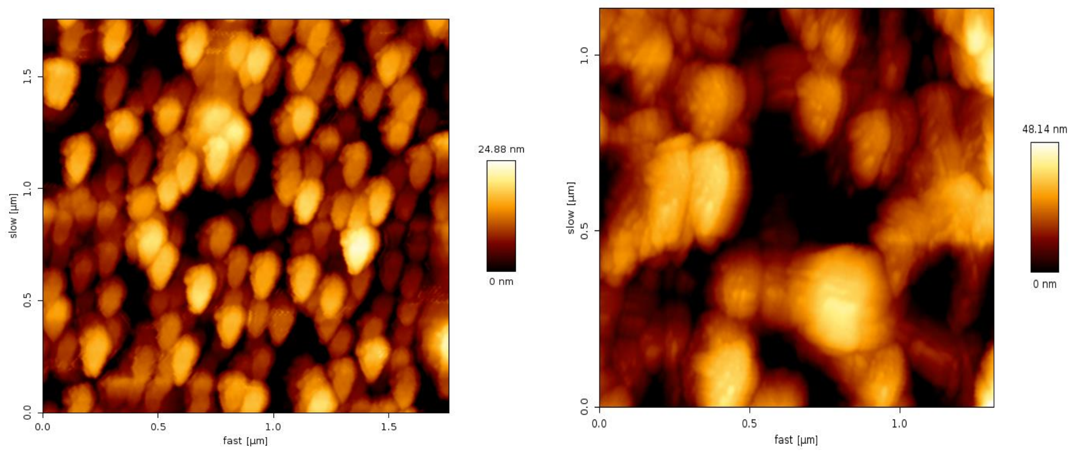

Figure 7: AFM images of (a) 0.3M/ m-Al$_2$O$_3$ and (b) 0.5M/ m-Al$_2$O$_3$.

3. 2. Release Profiles

To obtain the release profiles of drugs, a small number of drug-loaded carriers can be placed into a significant amount of release media via a dialysis bag. For the in-vitro drug release investigation, the ciprofloxacin drug was loaded into a 0.3M/m-Al$_2$O$_3$ carrier, and Eq. (2) was used to calculate the amount of drug loaded [22, 23]:

$$\text{Loading}\left(\frac{\text{mg drug}}{\text{g sample}}\right) = \frac{\text{m orig} - \text{m solu}}{\text{m MPS}} \quad (2)$$

where m_{orig} is the weight of ciprofloxacin (in milligrams) in the solution, m_{solu} is the weight of ciprofloxacin (in milligrams) in the solution after impregnation, and m_{MPS} is the weight (in grams) of the carrier sample.

The released amount of the drug was obtained from the following equation:

$$\text{Amount release} = \frac{\text{Ce} \times \text{V}}{\text{w}} \quad (3)$$

where C_e is the equilibrium drug concentration (mg. L^{-1}), w is the weight of the carrier (g), and V is the volume of the solution (L).

The release percentage of the drug (drug release%) was obtained from the following Equation:

$$\text{Drug release }\% = \frac{\text{amount of drug release}}{\text{amount of drug loading}} \times 100 \quad (4)$$

The determined amounts of ciprofloxacin loaded in the carrier were 2.82 milligrams of drug per gram of carrier. These loading values are consistent with the capacity of other adsorbents-drug defined by previous studies [34, 35].

In this study, drug release behavior was investigated at 37 °C, and pH= 7.4 and 5.4 (physiological condition). The cumulative ciprofloxacin release is shown in Fig. 8.

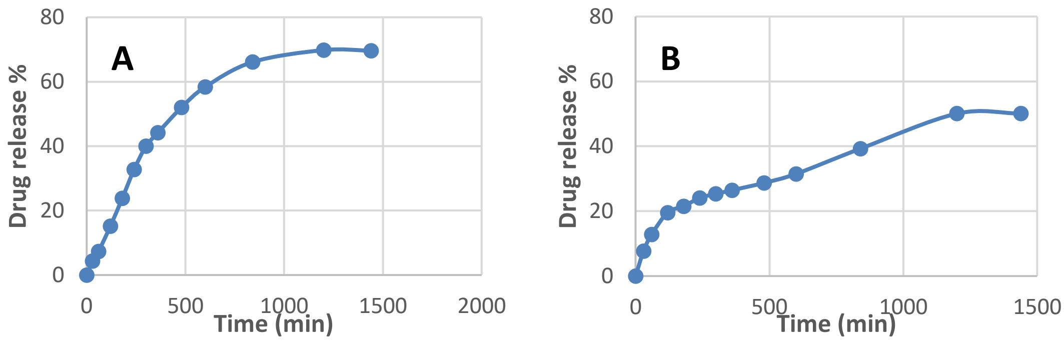

Figure 8: The release profile of ciprofloxacin drug loaded in 0.3M/m-Al_2O_3 at 37°C at pH: A) 7.4, and B) 5.4.

For the ciprofloxacin/m-Al_2O_3 system, 69.84% of loaded ciprofloxacin was released at pH 7.4, which decreased to 50.05 % at pH 5.4. The higher quantity of ciprofloxacin released at higher pH values was attributed to greater solubility in this condition [25, 26].

4. Conclusions

The current study prepared mesoporous alumina from two different concentrations of aluminium sulfate precursor by the microemulsion method. Based on the XRD patterns, the two prepared mesoporous alumina samples had identical cubic structures and exhibited perfect indexing of all diffraction peaks to the m-Al_2O_3 structure. BET surface area and BJH porosity results indicated that the 0.3M/m-Al_2O_3 sample has a higher surface area, pore diameter, and pore volume than the 0.5M/m-Al_2O_3 sample; the pore size distribution based on adsorption information showed a small range distribution for both samples. SEM analysis revealed that the morphology of the two samples consisted of small spherical particles aggregated into spherical agglomerates. However, the 0.3M/M/m-Al_2O_3 sample displayed smaller particles than the 0.5M/M/m-Al_2O_3 sample. EDX analysis results indicated that the percentage of aluminium oxide was high, at 95.5% and 97.3% by weight for the 0.3M/m-Al_2O_3 and the 0.5M/m-Al_2O_3, respectively, with a small percentage of carbon, which may be due to incomplete burnout of surfactants and impurities from the precursor. The loading and release results showed that the amount of ciprofloxacin loaded in the carrier was 2.82 milligrams of the drug per gram of carrier. At pH 7.4, 69.84% of the loaded ciprofloxacin was released, which decreased to 50.05% at pH 5.4 within 24 hours. In addition, it can be concluded that the microemulsion method is suitable for preparing m-Al_2O_3 with a high percentage and is the most effective carrier for the ciprofloxacin drug.

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Conflict of Interest

Authors declare that they have no conflict of interest.

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تحضير أكسيد الألومنيوم متوسط المسام كنظام توصيل لدواء السيبروفلوكساسين باستخدام طريقة المستحلب الدقيق

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الخلاصة

تم تحضير عينتين من أكسيد الألومنيوم متوسط المسامية (-γ) (0.3 مول/م-Al_2O_3 و0.5 مول/م-Al_2O_3) باستخدام طريقة المستحلب الدقيق، حيث استُخدمت كبريتات الألومنيوم كمادة أولية للألومينا. المواد الخام للمستحلب الدقيق هي كبريتات دوديسيل الصوديوم (SDS) كمادة خافضة للتوتر السطحي، و1-بيوتانول كمادة خافضة للتوتر السطحي مشاركة، وn-هيكسانول كطور زيتي. تم فحص العينات باستخدام المجهر الإلكتروني الماسح (SEM)، ومساحة سطح BET، ومسامية BJH، ومعادلة درجة حرارة الامتزاز-الامتصاص N2 عند درجة حرارة 77 كلفن. استُخدمت تقنيتا حيود الأشعة السينية (XRD) ومجهر القوة الذرية (AFM) لتوصيف هذه العينات. أظهرت النتائج أن العينتين لهما بنية طور متطابقة وفهرسة مثالية لـ γ- Al_2O_3-، تتميز عينة 0.3 مول/م-Al_2O_3 بمساحة سطح أكبر، 229.18 متر مربع/جم، وقطر مسام 5.59 نانومتر، وحجم مسام 0.32 سم3/جم، مقارنةً بعينة 0.5 مول/م-Al_2O_3 . كان شكل العينتين عبارة عن جسيمات كروية صغيرة متجمعة في تكتلات كروية، إلا أن عينة 0.3 مول/م-Al2O3 أظهرت جسيمات أصغر من عينة 0.5 مول/م-Al_2O_3، وكانت نسبة أكسيد الألومنيوم عالية، حيث بلغت 95.5% و97.3% وزنًا لكل من 0.3 مول/م-Al_2O_3 و0.5 مول/م-Al_2O_3 على التوالي. وكنموذج، استُخدمت عينة 0.3 مول/م-Al_2O_3 كحامل لتوصيل دواء سيبروفلوكساسين. أُجري التحميل بطريقة التشريب، بينما تم الإطلاق عبر كيس غسيل كلوي يحتوي على محاليل عازلة بمستوى pH 7.4 و5.4. تشير النتائج إلى أن العينة يمكن أن تُستخدم كحامل مناسب لدواء سيبروفلوكساسين.

الكلمات المفتاحية: مستحلب دقيق، الالومينا متوسطة المسام، سيبروفلوكساسين، توصيل الدواء، صورة تعريف الإطلاق.