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Condition Optimization for the Synthesis of Castor Oil Templated Mesoporous Silica

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ABSTRACT

The present work provides suitable conditions for the synthesis of mesoporous materials with improved porosity using an optimization technique. The proposed preparation method involves forming final products by varying the amount of castor oil, media, and template removal. The obtained samples were examined by TGA, XRD, DRIFT and nitrogen physisorption studies. It was observed that the porosity of the obtained samples was dependent on the conditions under which the materials were synthesized. All synthesized materials showed the Type IV adsorption-desorption isotherm features of mesoporous, regardless of the conditions utilized during the synthesis. The sample synthesized using 2.5 g of a castor oil template in acidic media and calcination as a template removal, displayed the highest BET surface area of 906 m^2*/g, pore volume of 1.44 cm*^{3}*/g and a pore diameter of 6.5 nm. When reflux was used as a template removal technique on materials synthesized under the same conditions, the surface area was 900 m*^{2}*/g with a pore volume of 1.4 cm*^{3}*/g and a pore diameter of 6.6 nm. Even though the latter sample exhibited less surface area, it is still better than the former sample since the template removal by reflux is a greener technique than the removal through calcination. XRD results showed that no peak was observed in low-angle implying that the materials lack long-range order of the pores. This large surface area synthesized mesoporous materials are potential adsorbents or catalytic supports. Therefore, the present study promotes the production of eco-friendly materials using castor oil, a biodegradable and renewable resource, as a pore-directing agent.*

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INTRODUCTION

The discovery of mesoporous materials around three decades ago was a significant development in the area of materials science (Costa et al., 2021; Fatima et al., 2025; Kausar et al., 2024; Kurji et al., 2023; Manzano & Vallet-Regí, 2025; Moya et al., 2024; Pinto et al., 2024; Wang et al., 2023). These materials, characterized by pore sizes between 2 and 50 nm, occupy an important niche between microporous (<2 nm) and macroporous (>50 nm) materials (Colomer, 2024; Jha & Jain, 2021; Pagar et al., 2018). Their unique porous structures have enabled various applications such as gas separation, biosensors, catalysis, optics, sorption media, and biomedical.

Mesoporous silica has attracted attention due to its amorphous properties that provide a degree of flexibility regarding the overall structural properties of the material. Moreover, silanol groups found on the surface of mesoporous silica provide many possibilities for functionalization and uses (He & Shi, 2011; Medina-Juarez et al., 2016). These materials come in various morphologies and dimensions, possessing a range of mesostructures such as lamellar, hexagonal, wormhole, and cubic while others have a disordered shape (Beck et al., 1992; Khurana et al., 2023; Mekuye & Abera, 2023; Soltani et al., 2020). Besides, various factors can be adjusted when synthesizing mesoporous materials, such as wall thickness, pore size, width, pore volume including the morphology (Feng et al., 2023; Silva et al., 2024). Mesoporous silica in particular, is renowned for its chemical and thermal durability, closely tied to its pore walls' thickness (Awoke et al., 2020; Bharti et al., 2015). Mesoporous silica materials are auspicious for many applications due to their advantageous chemical properties, thermal stability, and biocompatibility. The enormous surface areas and pore volumes of these materials are particularly well suited for use as sorbents and catalytic supports.

Meanwhile, the templates can be used in the synthesis of mesoporous materials as structure-directing agents leading to the formation of ordered materials (De Aguilar Pedott et al., 2021; Deng et al., 2012). Surfactant-templated sol-gel system is one of the most widely used methods to synthesize these materials (Beck *et al.*, 1992, Kresge, *et al.*, 1992). Studies indicate that mesoporous materials can be synthesized by tetraethyl orthosilicate (TEOS) in a basic medium (Choi & Yang, 2003), and in an acidic medium (Andriayani et al., 2023; Bharti et al., 2015; Pal et al., 2020) via sol-gel synthesis and in all approaches uses a template such as CTMABr, P123 (Jia-heng et al., 2017; Lu et al., 2022), or through salt-assisted synthesis (Teng et al., 2016; Yu et al., 2001; Zhong et al., 2009). The majority of research employs non-renewable templates to create mesoporous catalytic supports which has several disadvantages (Barrabino, 2011; Elimbinzi et al., 2020; Gajjela et al., 2010; Sun et al., 2011). The preparation processes often necessitate extreme pH conditions, elevated temperatures, and the use of toxic chemicals, leading to environmental harm and increased costs. Additionally, the removal of these templates typically involves calcination, which releases CO_2 and other hazardous gases, further exacerbating environmental concerns. Moreover, the reliance on non-renewable resources for template production raises sustainability issues, as these finite resources may become scarce or subject to price volatility.

Recent reports have highlighted the potential of renewable crude castor oil mixtures in producing micelle-templated silica supports with needed properties (López-Cuevas et al., 2017; Mgaya, 2012; Nyandoro & Elimbinz, 2022). Castor oil is a naturally occurring structure with a long alkyl chain, whose fatty acid chains are composed of approximately ninety percent (90%) ricinoleic acid, other components include linoleic acid (4.2%), oleic acid (3%), stearic acid (1.0%), palmitic acid (1.0%), dihydroxystearic acid (0.7%), linolenic acid (0.3%), and eicosanoic acid (0.3%). The ricinoleic acid, a monounsaturated 18-carbon fatty acid that constitutes the major component of castor oil, is an amphiphilic molecule due to its polar carboxylic and hydroxyl groups (the head) and its long nonpolar carbon chain (the hydrophobic tail). These fascinating attributes make castor oil a readily available natural template suitable for preparation of mesoporous materials. The materials obtained using castor oil showed some characteristics of mesoporous materials but still require improvement in surface area and pore diameter. The present study employs an optimization process to synthesize silica with superior mesoporous properties. The synthesis process involves the consumption of an eco-friendly bio template (castor oil) as a pore-directing agent. To achieve this, the synthesis was carried out in neutral, salt-assisted, and acidic conditions. Furthermore, the amount of template applied and the method of its removal were examined.

METHODS AND MATERIALS

Materials

Castor seeds were collected in Iringa region, Tanzania. The chemicals used include methanol, sodium fluoride, tetraethyl orthosilicate (TEOS), hydrochloric acid, *n*-hexane and ethanol, all purchased from Sigma Aldrich. The chemical reagents were procured from commercial sources and consumed without further purification.

Methods

Extraction of oil from the castor seeds

Three different approaches were employed during the extraction of castor oil namely; cold extraction, hot extraction (Soxhlet method) and mechanical extraction. These approaches were used to determine the method that yields more oil from the seeds. The castor seeds were sun-dried before the extraction procedure to simply the removal of the seeds from their coating. To extract the oil from the castor seed cake for cold and Soxhlet extraction, the castor seeds were ground further with a mortar and pestle. All extraction methods employed 1500 g of castor seeds. Ground castor seeds were soaked in 1.5 L of n-hexane for four days for cold extraction, after which the oil was filtered and concentrated under reduced pressure using a rotary evaporator at 40 °C, yielding 100 mL. On the other hand, the hot extraction was carried out using Soxhlet extraction, using 250 mL of n-hexane as the extraction solvent. During the extraction, 10 g of crushed seeds were placed in a thimble in the centre of the extractor, and n-hexane was heated in a round-bottomed flask at 60 °C, causing the *n*-hexane vapor to evaporate and pass through the thimble. The extract was condensed and collected in a round-bottomed flask for 6 h, yielding 2 mL of castor oil per 10 g of crushed castor seeds. The procedure was repeated several times to obtain sufficient oil. The solvent was removed under vacuum with a rotary evaporator, yielding 300 mL of castor oil. Castor seeds were crushed and pressed with an oil-pressing machine before being squeezed to yield 31.25 mL of castor oil from 1500 g of seeds.

Synthesis of silica materials using castor oil/water/ethanol

The synthesis of mesoporous materials was done as per the reported method with some modification (Elimbinzi & Mgaya, 2024; Mubofu et al., 2011). In a typical experiment, 2.5 g of castor oil was dissolved in a stirred mixture of 53 mL of distilled water and 47 mL of ethanol. The mixture was stirred at 35 °C for 2 h before adding 23 mL of TEOS, then stirred for another 24 h before aging at 80 °C for 24 h in a closed vessel, without forming a solid product.

Mesoporous silica materials synthesis using castor oil/water/ethanol/hydrochloric acid

Mubofu and coworkers’ method was adapted with some modification (Mubofu et al., 2011), where the mixture was then stirred at 35 °C for 2 h before adding 23 mL of TEOS and aging at 80 °C for another 24 h in a closed vessel. The synthesized product was filtered three times and washed with ethanol, then dried. The dried product was divided into two parts. The organic template was removed in the first portion (2 g) by Soxhlet extraction with 200 mL of ethanol as the extracting solvent during reflux at 70 °C for 6 h, and in the second portion (2 g) by calcination at 500 °C for 6 h. The resulting materials were named mesoporous templated silica MTS (R) and MTS(C), which stand for reflux and calcination, respectively. To increase the amount of materials, the synthesis was repeated three times, and the time for template removal in the reflux extraction was extended to 10 h. The castor oil is known to have a boiling point of about 300 °C however, in this study ethanol was used to dissolve the castor oil which refluxes at 70 °C. For the synthesis of the support materials, different sets of reactions were carried out using varying amounts of castor oil (2.5, 5.0, 10, and 15 g).

Synthesis of mesoporous silica materials using castor oil/water/hydrochloric acid

A stirred mixture of 308 mL of distilled water and 57 mL of 2 M HCl was used to dissolve 2.5 g of castor oil. The resulting mixtures were stirred at 35 °C for 2 h before adding TEOS (23 mL), which was then stirred at 35 °C for 24 h before being aged at 80 °C for another 24 h in a sealed vessel. During filtration, the synthesized product was washed three times with ethanol and allowed to dry overnight at room temperature. Soxhlet extraction with 200 mL of ethanol was used to remove the organic template after 10 h of reflux at 70 °C. After drying and filtering, mesoporous material was produced.

Synthesis of mesoporous silica materials using castor oil/water/ethanol, Sodium fluoride

Zhong et al (2009)'s method was adopted with some modification (Zhong et al., 2009), where 2.5 g of castor oil were dissolved in a stirred solution of 308 mL distilled water and 57 mL 2 M HCl. The resulting mixtures were stirred at 35 °C for 2 h before the addition of 23 mL of TEOS, which was then stirred at 35 °C for 24 h before being aged in a closed vessel at 80 °C for another 24 h. During filtration, the synthesized product was washed three times with ethanol before being dried overnight at room temperature. The organic template was removed using Soxhlet extraction, which involved refluxing 200 mL of ethanol at 70 °C for 10 h, resulting in mesoporous material after filtration and drying.

Materials Characterization

The Brunauer–Emmett–Teller (BET) surface area and the porosity measurements of the samples were studied by a nitrogen gas adsorption-desorption instrument (Quantachrome Instrument Nova 4200 multi-station nitrogen sorption system). The pore size distribution (PSD) and specific desorption pore volumes were obtained using the Barrett-Joyner-Halenda (BJH) method, and desorption branches were used to determine the PSD (Barrett et al., 1951; Fagerlund, 1973). Bruker AXS D8 Advance X-ray diffractometer (XRD) with nickel-filtered Cu Kα radiation (λ = 1.5406 Å) at 40 kV, 40 mA, at room temperature was used to analyze the diffraction patterns of the synthesized materials. The diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) studies were carried out using a Thermal Nicolet 6700 FTIR spectrometer packed with ATR under 10% helium flow and cooled with liquid nitrogen to analyze the synthesized materials' functional groups. Thermogravimetry analysis (TGA) of the synthesized mesoporous silica materials was carried out using the PerkinElmer Pyris 6 TGA thermal analyzer. 15 mg at a heating rate of 10 °C per minute to 800 °C in a nitrogen environment, with a purge rate of 20 mL/minute.

RESULTS AND DISCUSSION

Properties of oil extracted from castor seeds using different techniques

During the oil extraction process, several strategies were purposefully applied to determine the best technique for yielding a large amount of oil and providing materials with favourable qualities when the oil was used as a template. Using 1500 g of raw materials, Soxhlet extraction produced a higher yield of 300 mL than mechanical and cold solvent extraction, which produced 31.25 mL and 100 mL, respectively. In contrast to cold solvent and mechanical extraction techniques, a high yield was noted because the hot solvent passing through the ground castor seed allowed the oil to exit the seed with ease. Materials with similar surface area and pore size were synthesized from oils extracted using all available methods; however, mechanically extracted oil produced mesoporous materials with a lower surface area. This could be due to the oil's higher viscosity than the other oil extracted through solvent extraction. The template removal via Soxhlet had a lower surface area than when the calcination is used as the method of template removal. When compared to the calcination approach, template removal using Soxhlet was found to have a reduced surface area, which resulted in an increase in reflux time to 10 h.

Mesoporous silica materials development using castor oil/water/ethanol

Mesoporous silica was prepared using TEOS as a precursor in the mixture of castor oil, water and ethanol (TEOS:Co:C_2H_5OH:H_2O) in a neutral environment. The absence of a solid even after a full day of stirring suggests that the silica condensation process cannot take place in a neutral environment demonstrating that the synthesis of mesoporous silica requires basic or acidic conditions. Studies show that the synthesis of mesoporous silica was accomplished under similar conditions when cashew nut shell liquid was used as a template (Mubofu et al., 2011). Moreover, deeper investigation showed that only cardanol, one of the two main ingredients in cashew nut shell liquid, was discovered to be a useful template (Msigala & Mdoe JEG, 2012). In a neutral medium, the synthesis of mesoporous materials using castor oil failed due to the lack of driving force for the self-assembly, hydrolysis and condensation to take place. However, adding sodium fluoride to the reaction mixture resulted in the formation of the desired product. The addition of sodium fluoride as a catalyst improves the hydrolysis and condensation processes, both of which play important roles in the formation of mesoporous silica materials (Yu et al., 2001). In both cases, the mesoporous materials obtained were analyzed for surface area characteristics, with materials synthesized under acid medium (H_2O:C_2H_5OH:HCl) exhibiting a large surface area when 2.5 g of template was used.

Mesoporous silica materials synthesized using casto oil/water/ethanol/hydrochloric acid

In contrast to mesoporous materials synthesized in neutral conditions, the synthesis of mesoporous silica using the same template source (castor oil) was successful in an acidic medium (TEOS:Co:C_2H_5OH:H_2O:HCl) without the addition of sodium fluoride. The pH of the reaction mixture was between 3.7 and 4.2. Based on these findings and the reaction optimization conditions, it is possible to synthesize mesoporous silica using castor oil as a template source in acidic and basic mediums (Jia-heng et al., 2017; López-Cuevas et al., 2017).

Structural properties of mesoporous silica

Nitrogen gas adsorption-desorption studies at 77 K were used to investigate the textural properties of the synthesized MTS samples under various conditions. Generally, surface area and porosity of the synthesized materials are very important in surface science and heterogeneous catalyst design. The total surface area of a solid catalyst is critical as it determines the availability of active sites, which are key to catalytic activities. Similarly, pore architecture regulates transport phenomena in a heterogeneous catalyst, thereby determining selectivity during catalysis (Siles-Quesada et al., 2023; Storck et al., 1998). A typical BET plot, which is the plot of $1/[W/((P/Po)-1)]$ against P/Po, produces a straight line and is normally limited to relative pressures between 0.05 to 0.35

(Ahlquist, 1978; Bläker et al., 2019). The BJH model, which is built on the corrected Kelvin equation for multilayer adsorption can be used to calculate the pore size distribution and diameters.

Figure 1A–D shows the nitrogen gas physisorption results of the MTS samples synthesized at different conditions. Generally, the isotherms of all MTS samples exhibited Type IV isotherms with hysteresis loops centered at relative pressures above 0.4. The Type IV isotherms are characteristic of mesoporous materials and are normally associated with capillary condensation of the adsorbent that occurs in the mesopore (Blin et al., 2006). Multilayer adsorption on the pore walls always occurs before capillary condensation. Thus, in the early stages, type IV isotherms took the same path as type II isotherms. A sharp increase in adsorption indicates the onset of capillary condensation within the mesopore structure, which is consistent with previous findings (Park et al., 2012; Rana et al., 2011; Rani & Srivastava, 2017). In addition, all samples obtained in the present study revealed the hysteresis loops that are similar to type H2 according to the IUPAC classification systems demonstrating the presence of channel-like pores (Hilonga et al., 2010; Shao, 2024).

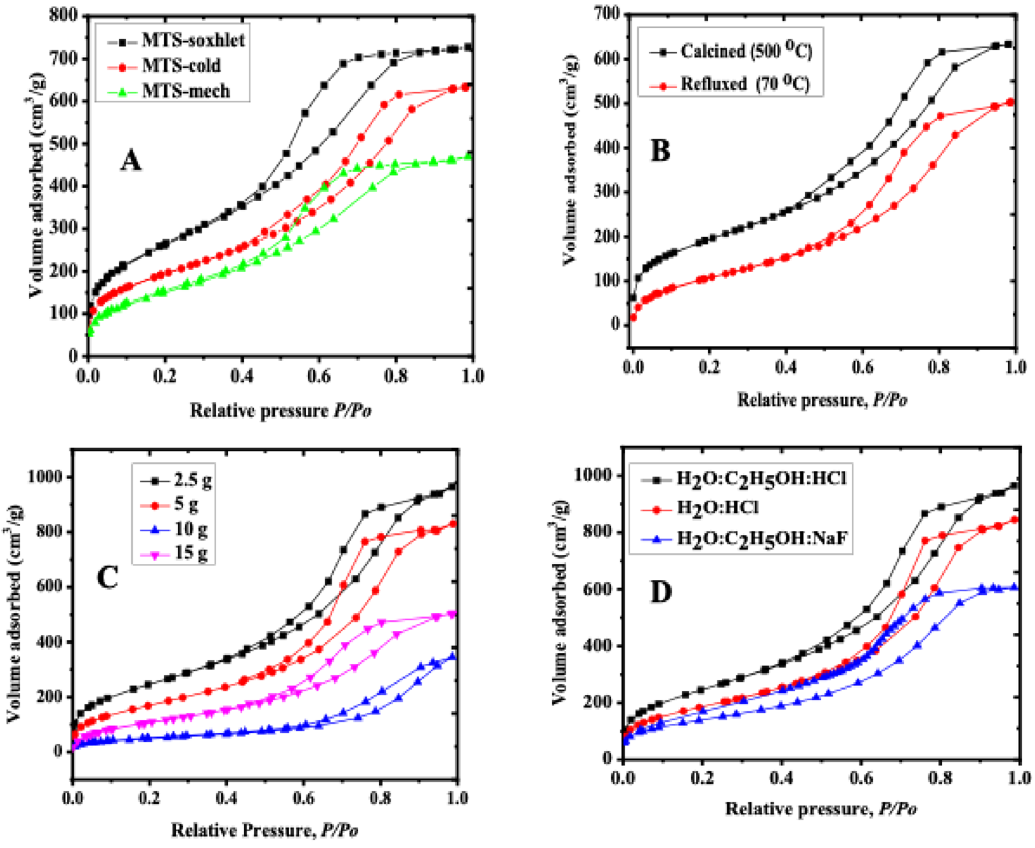

Figure 1: Nitrogen gas adsorption-desorption isotherms of the MTS samples synthesized using castor oil at different conditions: (A) using various extraction method; (B) using different template removal technique; (C) using different weight of castor oil; and (D) using different media.

Figure 1A shows that the samples synthesized with castor oil from various extraction methods (mechanical, Soxhlet, and cold) retained the type IV adsorption-desorption isotherms. Nevertheless, when mechanically extracted castor oil was used in the synthesis of the samples (MTS-mech), the sharpness of the adsorption isotherm decreased, most likely due to the oil's viscosity and thickness in comparison to solvent-extracted oil. This caused some oil to remain in the material after the template was removed. Figure 1B depicts the results obtained when a template removal was either through calcination or reflux. The effect of template removal on the adsorption-desorption isotherms was minor, with no discernible type change. Since the synthesized mesoporous materials' surface area and isotherm type resembled those of the calcination procedure, the reflux approach was hence employed in the present study to remove the template.

Evaluation of the amount of castor oil used as a templating agent on the textural properties of the obtained samples indicated that increasing the amount of castor oil during the synthesis did not affect the type IV features of the final products except for isotherm intensities as expected. On the other hand, changing the medium used during synthesis was also investigated, with the acidic medium using H_2O:HCl, H_2O:C_2H_5OH:HCl, and H_2O:C_2H_5OH:NaF for synthesis of MTS samples. Different media have been reported to be used during the synthesis of mesoporous materials (Pal et al., 2020; Sacramento et al., 2019). The various pH conditions used during the synthesis produced mesoporous materials with varying adsorption-desorption isotherm intensities. This could be due to the amount of N_2 gas adsorbed in the materials however, all samples maintained the type IV characteristics (Figure 1A-D).

Investigations were conducted into the castor oil extraction process as well as its impact on the surface area and pore size distribution of the produced mesoporous materials. The BET surface areas, pore volumes and pore diameters of samples obtained at different conditions are presented in Table 1. It can be seen that the BET surface areas exhibited by the samples varied between 292 m^2/g (low surface area) to 906 m^2/g (high surface area). Template removal on the surface was also tracked and compared as well. The removal by calcination method resulted in materials with a higher surface area of 906 m^2/g than that was removed by the reflux method (795.2 m^2/g). This could be because all the castor oil was completely burned at this high temperature, resulting in a larger surface area than when reflux was used as the template removal method. As a result, the reflux time to remove the template from the synthesized materials was extended from 6 h (795.2 m^2/g) to 10 h that resulted to the formation of final product with the surface area of 900 m^2/g. This indicates that the duration of reflux was found to have a significant increase in surface area. In comparison to alternative extraction techniques, mechanically extracted castor oil yielded a mesoporous substance with a small surface area. The small surface area of the material synthesized using mechanically extracted oil could be attributed to the oil's viscosity as compared to solvent-extracted oil.

The amount of castor oil utilized in the synthesis of the mesoporous material was assessed, along with its impact on the pore diameter, pore dispersion, and surface area. It was found that MTS samples with variable surface areas were produced when different quantities of castor oil were utilized. In comparison to other amounts, 2.5 g of castor oil provided the materials with a large surface area of 883.2 m^2/g. In Table 1, for example, using 15 g of castor oil reduced the surface area to 292.1 m^2/g. This can be explained by the fact that excess oil leads to a space between micelles, which prevents pores from attaching to one another. The surface area decreases with increasing surfactant load might also be attributable to inefficient silica condensation and potential pore collapse or blockage during template removal. As a result, 2.5 g of castor oil was chosen as the optimal weight for synthesizing mesoporous materials, which were then used as catalyst supports.

Table 1: Textural properties of synthesized mesoporous materials

Sample	BET surface area (m^2/g)	Total pore volume (cm^3/g)	Pore diameter (nm)
MTS	899	1.47	6.7
MTS-1	677	1.32	6.7
MTS-2	506	1.06	6.7
MTS-(Soxhlet)	783	1.17	6.7
MTS-(Soak)	729	1.01	6.5
MTS(Mech)	436	0.26	3.4
MTS(2.5 g)	883	1.37	6.8
MTS(5 g)	624	1.23	6.7
MTS(10 g)	378	0.53	5.7
MTS(15 g)	292	0.32	3.0
MTS(C)	906	1.44	6.5
MTS(R7)	795	1.32	6.4
MTS(R10)	900	1.40	6.6

The impact of various synthesis media used during synthesis on the surface area behaviour of as-synthesized materials was also investigated. As a result, three different media: $H_2O:C_2H_5OH:HCl$ (MTS), $H_2O:HCl$ (MTS-1), and $H_2O:C_2H_5OH:NaF$ (MTS-2) were tested for the synthesis of mesoporous materials as a catalyst support. Mesoporous materials synthesized with $H_2O:C_2H_5OH:HCl$ had a higher surface area of 898.8 m^2/g than the rest, which had 677.0 m^2/g and 505.8 m^2/g for $H_2O:HCl$ and $H_2O:C_2H_5OH:NaF$, respectively. MTS and MTS-1 synthesized under the aforementioned conditions had similar pore size distributions. However, they differed significantly from MTS-2, which had a pore size distribution with two peaks of lower intensity (Figure 2D).

Figure 2A-D depicts the pore size distribution of the samples synthesized with castor oil at different conditions. When compared to other extraction methods, mechanically extracted oil had a narrower pore size distribution and a shorter peak. Figure 2B reveals that template removal methods, reflux, or calcination had no effect on the pore size distribution of the synthesized MTS samples. Researchers reported mesoporous materials with high surface area using different template (Jambhulkar et al., 2020; Shinde et al., 2021; Zhao et al., 2012). Nevertheless, the use of castor oil offers an advantage over petroleum-based surfactants commonly used in earlier studies since it is renewable and biodegradable resource (Liu et al., 2010; Watthanachai et al., 2019). It was found that the amount of castor oil used in the synthesis process had no effect on the pore size distribution. For example, increasing the amount of castor oil in the synthesis procedure from 2.5 g to 15.0 g resulted in negligible variation in pore size distribution (Figure 2C).

However, the pore diameter and volume decreased as the amount of castor oil used in the synthesis increased especially when 15 g of castor oil used. In addition, the pore diameter remains constant that might be ascribable to the stable micelle sizes. However, there is a decrease in the average pore size for different amounts of castor oil used observed. For example, the pore diameter decreases from 6.8 nm to 3.0 nm when 2.5 g and 15 g of castor oil are used, respectively. Depending on the synthesis conditions, the produced mesoporous silica materials showed distinct pore architectures with pore sizes varying from 3 to 6.8 nm.

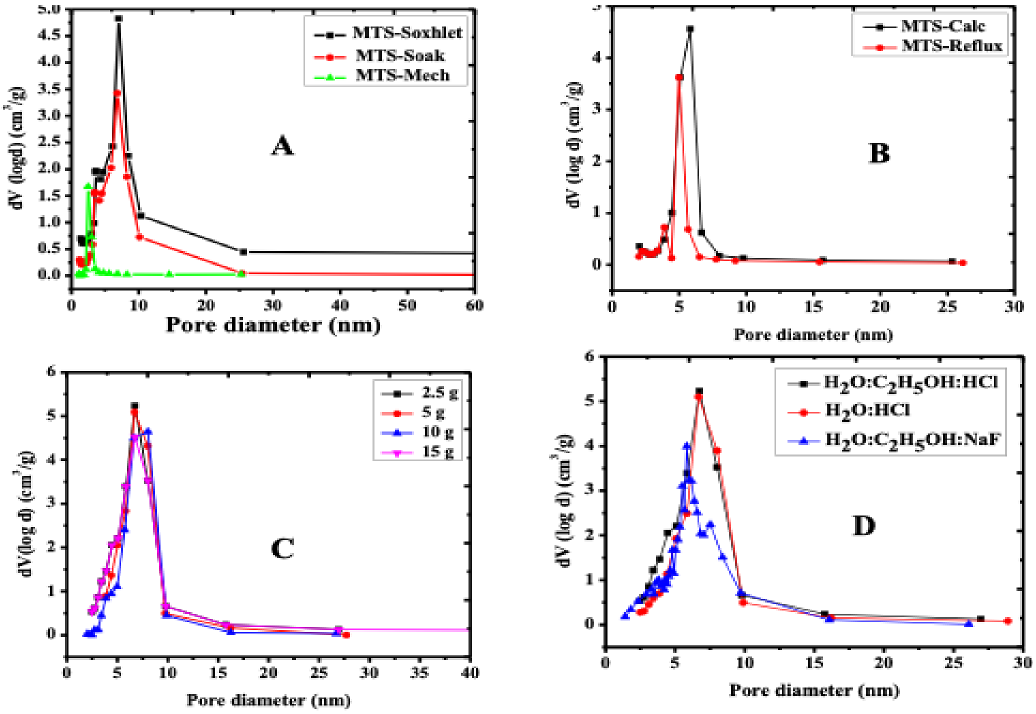

Figure 2: Pore size distribution of MTS samples synthesized with castor oil at different conditions: (A) using various extraction method; (B) using different template removal technique; (C) using different weight of castor oil; and (D) using different media.

The BET surface area ranged between 292–906 m²/g, and pore volumes were in the range of 0.32–1.47 cm³/g. The results are comparable or superior to similar studies utilizing bio-based templates as pore directing agent. For instance, Mgaya (2020) reported BET surface areas of 428–605 m²/g, pore diameter 8.5-8.9 nm and pore volumes of 1.0 – 1.1 cm³/g for mesoporous silica synthesized using castor oil, while Jovita et al. (2024) observed BET surface areas of 439-541 m²/g, pore diameter ranging from 6.08 to 6.55 nm with pore volumes of 0.48 – 0.58 cm³/g when using Sapindus rarak extract as template (Jovita et al., 2024; Mgaya, 2020).

The low angle and wide angle XRD analyses were used to further study the structural properties of the mesoporous templated silica materials synthesized using castor oil as templates. Figure 3A shows that there was no peak in the low-angle XRD diffractograms for all batches of mesoporous silica materials signifying that the synthesized materials lacked a broad range of pore order as was revealed by the nitrogen gas physisorption studies. Synthesis of mesoporous materials lacking long-range of pore order has been reported by other researches (Alothman, 2012; Narayanaswamy et al., 2008; Sharma et al., 2023). When the same materials were characterized using wide-angle XRD, a broader peak was observed indicating the disorders and presence of amorphous silica (Figure 3B-D). Similar observation was reported by different scholars (Elimbinzi & Mgaya, 2024; Okoronkwo et al., 2016; Purnawira et al., 2019). It is noteworthy that the XRD patterns of the samples were unaffected by the synthesis conditions, as evidenced by a broad peak seen in all synthesized materials under various conditions (Figure 3B-D).

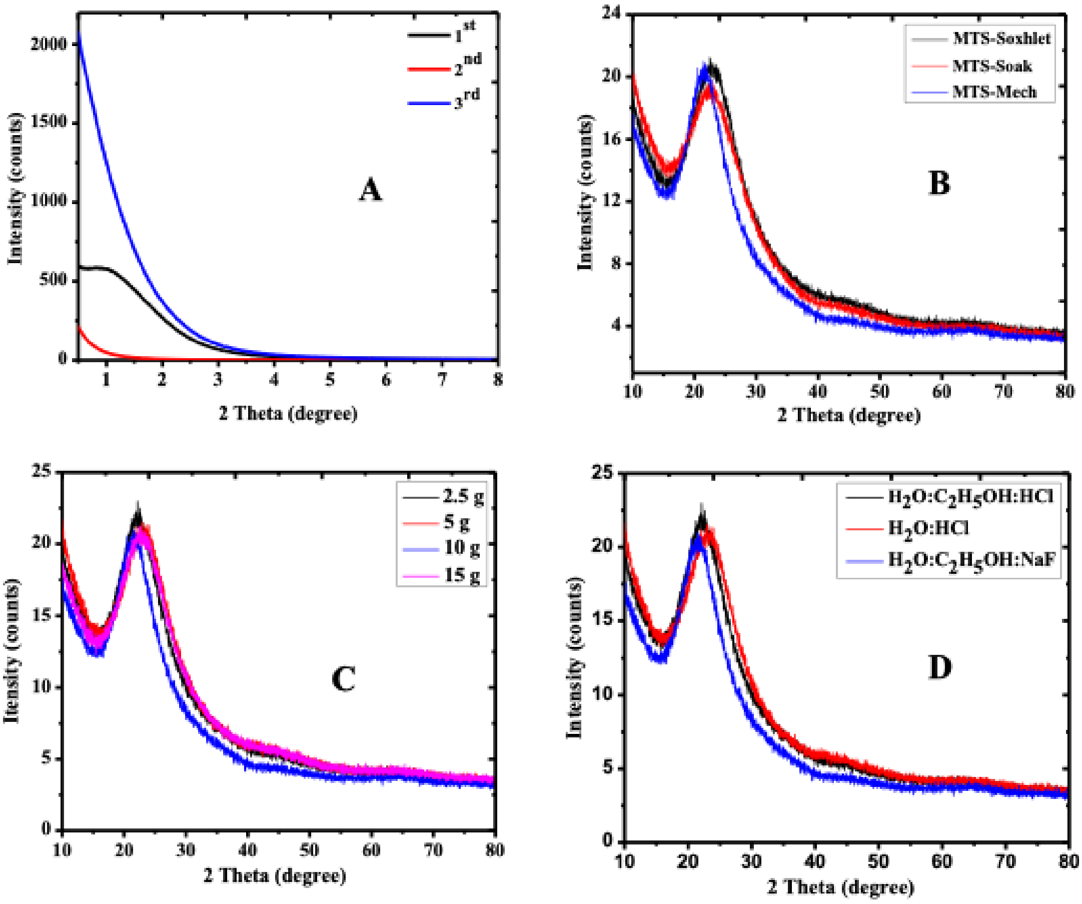

Figure 3: XRD patterns of the MTS synthesized using castor oil at different conditions; (A) Low angle XRD patterns (B) Wide angle XRD patterns MTS synthesized using castor oil extracted from different methods; (C) Wide angle XRD patterns of MTS synthesized using different amount of castor oil; and (D) Wide angle XRD patterns of three different conditions of the synthesized MTS.

DRIFT was utilized to determine the anchoring functional groups on the produced materials. All spectra for synthesized materials revealed the same functional groups despite being synthesized under different conditions. Figure 4 shows the DRIFT spectra of mesoporous templated silica samples synthesized via various conditions.

The spectra were drawn by picking representative from different condition to show functional groups formed. The presence of Si-O-Si in the spectra of the samples shows silane vibrations were observed around 1067 cm^{-1} which is also associated with absorptions at 3569 and 970 cm^{-1}, indicating the presence of Si-OH (Elimbinzi et al., 2018). Various silica vibrations were observed in the range 1000 -1260 cm^{-1} indicating the presence of silica species, with Si-O-Si symmetric observed at 800 cm^{-1}. In addition, the Si-O-Si stretch of silica observed around 1102 cm^{-1}, 1645 cm^{-1} and 3456 cm^{-1} were assigned to surface hydroxy groups of mesoporous silica (Alahmadi et al., 2012) and Andriayani et al (Andriayani et al., 2023).

Meanwhile, study investigated the thermal stability of the mesoporous silica samples synthesized under different conditions. Figure 5 shows TGA plots for mesoporous templated silica under various conditions. The weight loss below 100 °C due to physiosorbed water and additional weight loss above 600 °C due to silanol group condensation as observed by other researchers (Imoisili et al., 2020; Nyandoro & Elimbinz, 2022). Generally, the TGA and DRIFT results revealed that all of the synthesized mesoporous materials obtained materials had stable structure and thermally stable as well.

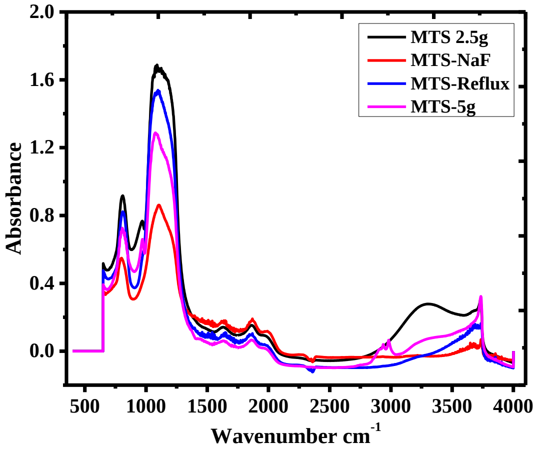

Figure 4: The DRIFT spectra of mesoporous templated silica synthesized via various condition

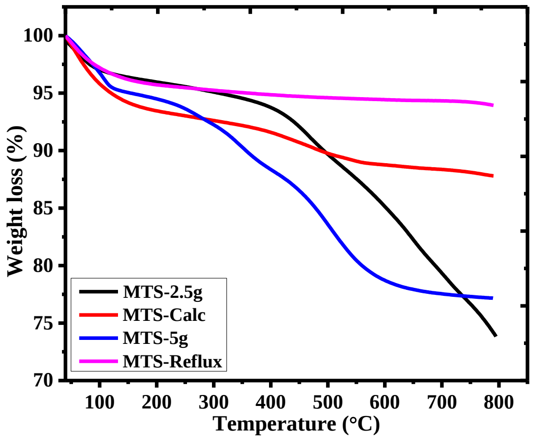

Figure 5: TGA profiles of MTS synthesized under different conditions.

The present study revealed that the morphology of silica can be controlled through consumption of castor oil as a template to act as a structure directing agent. The formation of a porous materials is also dependent on the precursors and synthetic conditions (Lofgreen & Ozin, 2014). The role of castor oil as a structure directing agent has been previously reported (Jovita et al., 2024; Mgaya, 2020). When introduced into the reaction system containing inorganic precursors (TEOS), these molecules self-assemble to form stable micelles that shield the hydrophobic tails from the water, acting as a scaffold. The inorganic precursors then condense around the polar surface of these organized micelles. During the removal of the castor oil template, it leaves behind an inorganic solid with uniform, interconnected mesopores whose structure directly mirrors the original micellar arrangement (Jovita et al., 2024; Mgaya, 2020). This makes the castor oil to be considered as effective and green structure-directing agent for formation of mesoporous materials as was exemplified by the nitrogen physisorption studies results. Therefore, the designed synthetic conditions to prepare MTS materials with desirable properties for catalysis using biodegradable and renewable template is commendable.

CONCLUSIONS AND RECOMMENDATIONS

The determination of the suitable conditions during the preparation process of mesoporous silica is very critical to facilitate the formation of thermally stable and porous structure. A variety of factors were examined, including the effect of the amount of castor oil employed, the media used during the synthesis, template removal, and the techniques for oil extraction during the preparation of mesoporous silica. Findings indicated that different surface areas were obtained as a result of variations of the preparation conditions. Structural characterization indicated that amorphous, thermal stable and porous silica can be obtained through the optimization technique. Specifically, the samples synthesized in an acidic medium (H_2O:C_2H_5OH:HCl) using 2.5 g of Soxhlet-extracted castor oil as a templating agent were considered to be ideal for the synthesis of MTS since they yielded final products with highest surface area and pore size distribution. The obtained results could be due to the high purity of the castor oil extracted via Soxhlet method which promote the process of self-assembly of micelles compared to the castor oil obtained via soaking and mechanical method of extraction which was impure.

The consumption of renewable templates like castor oil is highly recommended as it is effective in addressing environmental and economic concerns as it can replace conventional, non-renewable surfactants. Therefore, the present work provides a clear pathway for sustainable production of high-quality mesoporous silica materials. It is anticipated that the present work will stimulate the extension of these findings toward scale-and eventually evaluate the application of the resulted materials in various applications such as catalysis, adsorption, and biomedical fields.

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