(Liu et al., 2005, Panek et al., 2017) Different

types of low cost materials that have been used

as alternative sources of silica and alumina in

mesoporous materials synthesis (Xie et al,

2014) which was proven to be a way to

decrease the costs involved in the synthesis processes. However, pure siliceous mesoporous

MCM-41 materials are limited to extensive

applications for absence of acid sites and ion-

exchange capacity. the introduction of metal

ions doping into framework of MCM-41

materials, such as aluminum, iron, boron,

gallium and other transition metal elements $(Al^{3+}, Fe^{3+}, Ga^{3+} and B^{3+} e.t.c)$ can overcome

those obstructions (Wang et al, 2002). The



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SYNTHESIS AND CHARACTERIZATION OF AI-MCM-41 SUPPORTED ON CaO WASTE OBTAINED FROM WELDING SITE

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ABSTRACT

The utilization of CTAB (Hexadecyl trimethyl Ammonium Bromide) as a template in the synthesis of Al-MCM-41 was accomplished through a direct, uncomplicated onestep synthetic approach. To maintain a pH range of 10-11, the solution was subjected to a crystallization temperature of 100 °C for a duration of 24 hours. Following the crystallization process, the material was calcined in air for five hours at 550 °C. The incorporation of CaO nanoparticles, a byproduct resulting from the reaction of calcium carbide and water in the welding industry, into the Al-MCM-41 framework was carried out successfully. The effects of the incorporation are evident in the porosity, structure, and surface area of the AI-MCM-41 mesoporous materials. The material was characterized by X-ray powder diffraction, N2 adsorption-desorption, transmission electron microscopy (TEM) techniques, an Xray fluorescence, and Fourier transform infrared spectroscopy (FT-IR). Pure Al-MCM-41 displayed high ordered mesoporosity with a large surface area, and a large pore volume. However, the incorporation of CaO in the framework resulted in the surface area decreases from 765 to 512 m²g⁻¹, the pore volume also decreased from 0.8 to 0.1 cm³g⁻¹, and the average diameter increased from 18.5 to 18.44 nm. The basicity of the catalyst was found to be optimum at 20% loading CaO. The resulted, CaO/Al-MCM-41 might be produced on a large scale and serve as catalyst for chemical transformation.

Keyword: Hydrothermal, mesoporous, thin sheet, Calcium oxide

INTRODUCTION

Mesoporous materials, especially MCM-41 materials, have been widely used as adsorbents, molecular hosts and catalyst carrier for their highly uniform channel and high surface area (Hamzehloo et al. 2018; Kocaman et al., 2018). The traditional synthesis of this type of ordered mesoporous materials is based on the hydrothermal route that uses an organic source of silica, such as tetraethyl orthosilicate (TEOS) and a surfactant, which plays the role of the structure directing agent, and that must be removed after the silica condensation so that the mesopores become available (Sterczynska et al., 2017; Bhattacharyya *et al.*, 2006; Alothman, 2012; Abu-Zied *et al.*, 2015) Such reagents are usually expensive and the MCM-41 materials built with pure-silica are of limited use for some applications, because of the lack of acid sites and poor ion-exchange capacity. Kang et al, (2005). Several studies have been devoted to the search of cheaper raw materials that could overcome the high costs associated with the synthesis of MCM-41.

enhancement of the siliceous framework, its structural characteristics, and surface reactivity has been demonstrated by On *et al*, (2003). In situations where the oxygen coordination number is constant, the introduction of a hydroxyl group results in the generation of a hydroxyl on the aluminum ion, thereby maintaining electrovalence balance. This process produces a Bronsted acid center, which

augments the chemical activity of MCM-41.So aluminum doping into MCM-41 is the most popular one of the modified works. The catalytic activity was enhanced by the incorporation of a basic or amphoteric oxide into the silica gel, whereas the reverse result was obtained when acidic oxides were deposited (Ai and Bull 1991). Recently, the mesoporous molecular sieve MCM-41 was developed and applied in many acidcatalyzed reactions (Zhao *et al*, 1996). However, up to now much fewer works on its role as a base catalyst have been reported (Jaenicke *et al*, 2000, Corma, *et al*, 1998).

In this work, AIMCM-41 was synthesized as a heterogeneous base catalyst via hydrothermal process and incorporation of CaO on the framework was studied.

Experimentation MATERIALS

Materials used in this work include waste calcium oxide from welding site, hot plate with a magnetic stirrer and mercury in glass thermometer, furnace, dry-oven and separating funnel, the chemicals such as acetic acid, sodium hydroxide and Hexadecyltrimethyl Ammonium Bromide (CTAB) were of analytical grade.

Preparation of CaO

Calcium oxide waste from the welding site was rinsed with distilled water before being dried at 105 °C. It was then mashed using a mortar after being crushed calcined at 900 °C for 5 h in a furnace to remove carbon remain and H_2O . (Yu, J 2001).

Preparation of Al-MCM-41

CTABr (2.0419 g, 99.0%, KERMEL) was dissolve in (20.0 g) distilled water to give solution 1. Solution 2 was then prepared by mixing aluminum chloride (1.6078 g, 95%) and sodium silicate (6.7722 g, 22.8% SiO₂) in distilled water (20.0 g) . Then, Solution 2 was added into Solution 1 with stirring before sodium hydroxide (0.4437 g, 5%) was added into the mixture. The precursor with a final molar composition of 4SiO2:0.2Al2O3:1CTABr:1Na2O:400H2O was hydrothermally treated at 100 °C for 24 h. The solution was cooled to room temperature and the pH of the solution was adjusted to 10-11 by adding 5% acetic acid. The heating and the pH adjustment procedures were repeated twice before the solid was purified, dried and calcined at 550 °C for 5 h (Ng et al., 2019).

Preparation of CaO/Al- MCM-41

5% of calcium oxide was dissolved in 30 ml distilled water, then 1 g of Al-MCM-41 was added with stirring. After drying in a desiccator,

the mixture was heated at 110 ^oC for 12 h and finally calcined at 500 ^oC for 4 h. The procedure was repeated for 15% and 20% calcium oxide (Yu, J 2001).

Characterization

Powder X-ray diffraction (XRD) patterns were collected on a Bruker-AXS D8 diffractometer with Cu Ka radiation (λ =1.5418 A). FT-IR spectra were recorded with a Perkin Elmer's System 2000 FTIR spectrometer, using the KBr method. Transmission electron microscopy (TEM) images were captured using a PHILIPS CM-12 TEM with an acceleration voltage of 200 kV. TGA/DTG analysis was performed using a Mettler TGASDTA851 instrument at a heating rate of 20 °C min⁻¹ under air flow. N₂ adsorptiondesorption isotherms were measured at -196 °C on a Micromeritics ASAP 2010 instrument. The solid was degassed for 15 h at 300 °C before analysis. 29Si Single Pulse Magic Angle Spinning (SPE-MAS) experiments were performed at a spinning rate of 12 kHz, using a pulse angle of $\pi/6$ (2.3 µs) and a recycling delay of 80 s under high-power proton decoupling conditions (63 MHz). The spectra were recorded at room temperature with a Bruker Avance II 300 MHz spectrometer equipped with a Bruker 7mm double channels probe. The analysis was operated at B0=7.2 T (Larmor frequency ν 0=59.62 MHz). The chemical shifts were quoted relative to tetramethylsilane (TMS). The deconvolutions of the spectra were performed by using the DMFit software. The 27AI MAS NMR spectra were recorded at room temperature B0=9.4 operating at Т. Durina the measurement, a Bruker 4mm double channels probe with a $\pi/12$ rf pulse length of 0.5 ms, a recycle delay of 1 s, at a spinning rate of 12 kHz, were used. The chemical shifts were referred to external [Al(H₂O)₆]³⁺ in AlCl₃ aqueous solution (Othman, et al., 2017).

RESULT AND DISCUSSION

Figure 1 shows the FTIR spectra of the wellwashed, unclaimed, and calcined solid samples with 5% CaO loading. The IR bands at 2921, 2853, 1479, and 788 cm⁻¹ demonstrate that the solids include an occluded CTABr template (Khoo *et al.*, 2013). As can be seen, compared to its two counterparts, the uncalcined AIMCM-41 exhibits the strongest bands, indicating that it has the most occluded CTABr template. Adsorbed water's -OH group is in charge of the components at 3461 and 1649 cm⁻¹, respectively (Berger *et al.*, 2015). These two bands were thinner when more Al atoms were incorporated into the framework of MCM-41, indicating that

the amount of water adsorbed. Additionally, the Al-MCM-41 exhibits a weak band at 952 cm⁻¹ that is associated with the bending vibration mode of isolated Si-OH groups (Fig.1b) (Goh *et al.*, 2013). The FTIR signals at 1232 and 1065 cm⁻¹, respectively, indicate the internal and external asymmetric stretching vibrations of the Si-O-T bonds (where T is either Si or Al) (Majano *et al.*, 2011). The latter band shifts as more CaO atoms are added to the AlMCM-41 siliceous framework, as seen Fig 1c. Therefore, it seems that the longer Si-O-Al connections in the

AlMCM-41 framework are causing the pore size to increase (Oxtoby *et al.*, 2002). The symmetric Si-O-T bending vibration is what causes the band to appear at 719 cm⁻¹, while the tetrahedral Si-O bending mode resonates at 453 cm⁻¹. The presence of the band at 2180 and 2020 cm⁻¹ implies that CaO has been incorporated into the AlMCM-41 frame work (Fig. 1c). This indicated that, aluminosilicates and CTABr combined to form AlMCM-41, and CaO is fully incorporates in to the framework.



FIG 1: IR spectra of (a) Uncalcined Al-MCM-41 (b) Calcined Al-MCM-41 (c) 5% CaO/ Al-MCM-41

The FTIR spectroscopy data were further supported by TG/DTG analysis (Fig. 2). Three steps of weight loss were clearly observed for the sample. The first step of weight loss at 100 °C was due to the removal of physisorbed water (Braga *et al.*, 2011). As seen, the amount of water adsorbed was reduced as the Al content increases in line with the FTIR spectroscopy data. The second weight loss at 350 °C was ascribed to the decomposition of occluded template *via* Hofmann elimination. As

shown, the amount of occluded CTABr during the formation of Al-MCM-41 framework. Third step of weight loss at 500 °C is due to the oxidation of the remaining organic components to carbon dioxide, water and probably residual carbonaceous species (La-Salvia *et al.*, 2017). At this step, small quantity of water is also produced from Si–OH condensation forming siloxane (Si–O–Si) groups (Pirouzmand *et al.*, 2017).



FIG 2: TG Spectra of calcined AI-MCM-41

Structural, morphological and porous characterizations

The occluded CTABr was taken out of the Al-MCM-41 nanoparticles by calcination, and their XRD patterns are displayed in **Figure 3**, Two small peaks at 2θ =4.11° and 4.78°, which can be attributed to the (100), (110) and (200) planes of the Al-MCM-41 type structure, respectively, are seen in the pattern for pure siliceous Al-MCM-41. According to this, a highly ordered Al-MCM-41 mesophore has successfully

been synthesized using a less expensive silica source and NaOH as a mineralizer, which is in accordance with the thermogravimetric analysis (Kresge, *et al.*, 1992). However, as more CaO molecules are deposited into the Al-MCM-41 framework, the XRD peaks weaken, showing the mesostructures long-range order has been disturbed (Fig. 3b, c and d). Furthermore, the growth of d100-spacing values causes these diffraction peaks to shift to lower 2θ angles (**Table 1**).



Fig 3: XRD patterns of calcined (a) Al-MCM-41, (b) 5%CaO/Al-MCM-41, (c) 15%CaO/Al-MCM-41 and (d) 20%Al-MCM-41

The peak intensities appearred to be decreasing due to the CaO deposit on AI-MCM-41, the sample does, however, still have the typical hexagonal structure. Despite the similarity in pore diameters, the blocking of pores was what caused the considerable reduction in BET surface area and pore volume that occurs during CaO deposition also the intensity of the XRD peak decreases.

Samples	Si_2O/Al_2O_3	d ₁₀₀ spacing	surface	pore	pore
		(nm) ²	area,S _{BET} (m ² g ⁻¹)	volume,V _{total(cm} ³ g ⁻¹)	diameter(Å) ^b
Al-a	∞	3.94	765	0.80	18.51
5%Al-a	5	3.78	624	0.47	18.46
15%Ala	15	3.46	591	0.21	18.45
20%Ala	20	3.10	512	0.10	18.44
	a =	MCM-41			

Table 1:Texture properties of Al-MCM-41 sample	es.
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Table 1 lists BET surface area, base amount, and catalytic results over various catalysts. As the amount of CaO deposited on Al-MCM-41 was increased, the catalyst base amount also increased markedly but with concomitant decrease in the surface area. Although Al-MCM-41 exhibits catalytic activity, its performance was significantly enhanced by the deposition of CaO. Furthermore, the catalytic activity correlated well with the catalyst base amount. The enhanced basic character of CaO on Al-MCM-41 was also evidenced by other base catalyzed reactions.

The surface morphology of the AIMCM-41 synthesized in this work was taken by TEM and the TEM micrograph is showed in Fig. 4. The TEM images of AI-MCM-41 catalysts with various CaO loadings are shown in Figure 4a. These images are exemplary TEM images of the calcined AI-MCM-41, which exhibited a highly organized hexagonal thin sheet structure. With an average size of 20 nm, pure siliceous AI exhibited a hexagonal particle size distribution (Fig. 4a).

It has a homogeneous shell thickness and a thin sheet structure with uniform mesopores at the surface that exhibits a hexagonal morphology. Similar morphology was also seen in Al-M-5%, where a distortion in the morphological structure of the particle due to a modest decreased in structural orientation of the thin sheet and thickness of the thin sheet. (Fig. 4b) However, the shape and the thin sheet were deteriorated due to the CaO level raised. However, the structural deterioration was more observed in sample containing 15 and 20% CaO nano particles. The findings supported the FTIR, TG/DTG, and XRD data, which demonstrate that AI-M-15% and AI-M-20% have considerably denser nanoparticle compositions due to their lower levels of mesoporosity.



Fig 4: TEM images of (a) Al-M-∞, (b) Al-M-5%, (c) Al-M-15% and (d) Al-M-20%

The AIMCM-41 synthesized showed typical type IV isotherm (Figure 5a) of MCM-41 mesoporous materials with two-dimensional hexagonal structures,. Due to multilayer adsorption, the amount adsorbed at the adsorption branch gradually rises as the relative pressure rises. It is possible that the instability of the liquid nitrogen meniscus inside the small channels was what caused the small hysteresis loop that was detected at relative pressures of 0.25 to 0.4 P/P₀. No noticeable hysteresis loop was seen at a relative pressure of 0.9, but a rapid step rise in the isotherm of the calcined sample at P/P_0 0.25 to 0.40 shows both the sample's uniform pore size distribution and its well-ordered mesoporous structure. The CaO-Al-MCM-41 sample (Figure 5b) keeps the same isotherm shape, but there was a reduction in the amount of nitrogen that was adsorbed, and the beginning of the capillary condensation step occurs at a lower relative pressure. The decreased pore size, which was

linked to the pore-filling action due to the integration of the CaO nanoparticles into the mesopores of the solid materials was what causes the shift of the inflection point of the step to lower relative pressure P/P_0 and the decrease in absorption quantity. The presence of comparatively homogenous mesopores in the samples is shown by the pore size distribution (Figure 5a, b) computed from the adsorption branches of the isotherms using the BJH model. While the pore volume and pore diameter reduce from 0.80 mL/g and 18.51 nm to 0.160 mL/g and 17.79 nm, respectively. The BET surface area decrease from 765 m²/g of Al-MCM-CaO/Al-MCM-41. 41 to 512 m^2/q of Comparatively, the Al-MCM-41 sample (Figure 5d) nearly loses the isotherm shape and uniform pore distribution, indicating that the CaO nanoparticles blocked the matrix's mesopores. This explanation is comform with the XRD data analysis (Fig. 3).



Fig 6: N2 adsorption (close symbol) and desorption (open symbol) isotherms, and pore size distribution (inset) of calcined (a) Al-M- ∞ , (b) Al-M-5%, (c) Al-M-15% and (d) Al-M-20% nanoparticles.

Table 2 shows the XRF formation of Al-MCM-41 and different samples loaded with CaO at different percentages i.e 5%, 15% and 20%, respectively. The Si/Al ratio experienced a discernible reduction consequent to the assimilation of CaO into the framework. The basicity of the specimens was assessed through the utilization of the back titration method. The 20%CaO/Al-MCM-41 specimen exhibited the most pronounced basicity (300 μ mol g⁻¹) owing to the elevated quantity of basic sites. In contrast, the Al-MCM-41 which is made up of pure siliceous framework recorded the lowest basicity (20 μ mol g⁻¹). It was because the number of CaO responsible for the formation of active basic site is absent as compared with 15 and 20% CaO/ Al-MCM-41 samples.

Table	2: XR	F Form	ation
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	XRF spectroscopy analysis				Si/Al molar		
Samples	SiO ₂ (%)	Al ₂ O ₃ (%)	CaO (%)	Others (%)	Total (%)	ratio	Basicity (µmol/g)
Al-a	94.43	1.83	N/A	0.25	100.00	91.68	20
Al-5%	92.82	2.02	0.13	0.33	100.00	80.96	180
Al-15%	91.90	2.47	0.75	0.36	100.00	65.94	250
Al-20%	90.12	2.63	0.92	0.41	100.00	60.43	300

CONCLUSION

The Al-MCM-41 was successfully synthesized by one-step method using silica-aluminum sources, and the structural and properties of these mesoporous materials measured by various analytical methods. Under the condition of pH 10-11, The Al-MCM-41 mesoporous material crystalizes at 100 °C or 24 h and calcination in air at 550 °C or 5 h. The one-step hydrothermal synthesized Al-MCM-41 mesoporous material has higher BET surface area, a lager pore size and pore volume. Moreover, Al-MCM-41 molecular sieves presented uniform size, thin sheet

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