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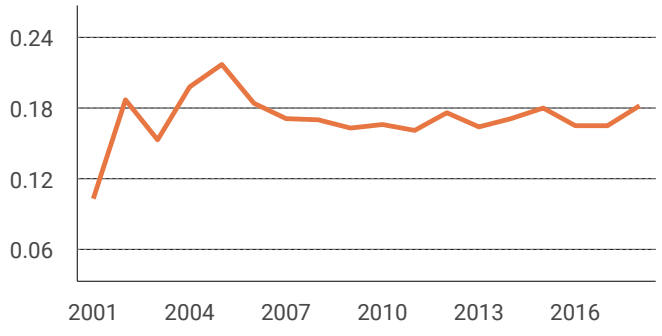

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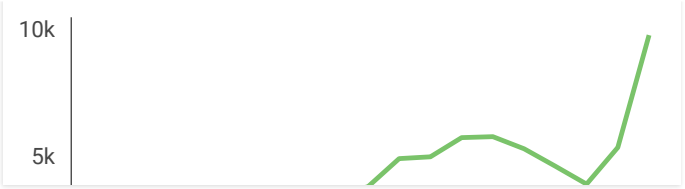

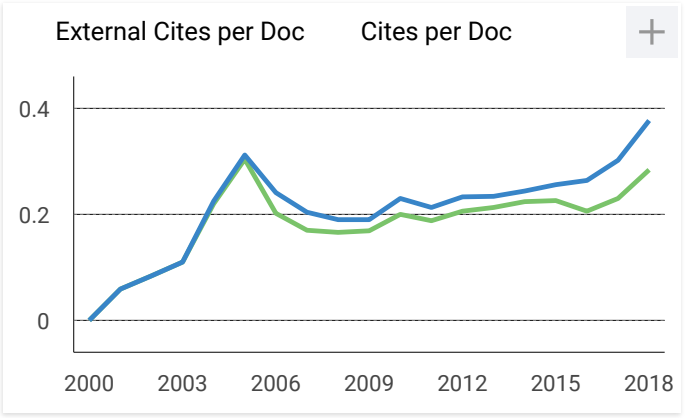

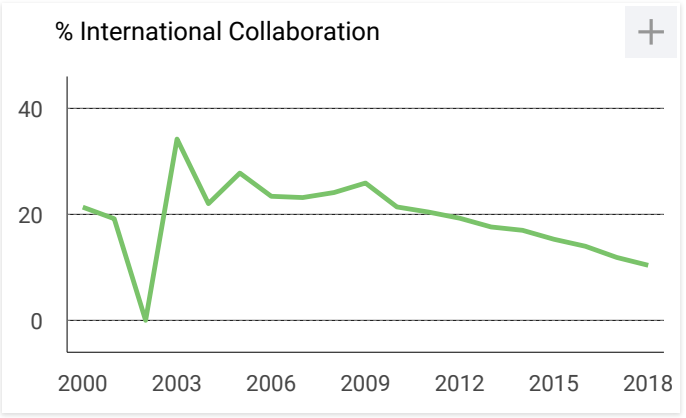

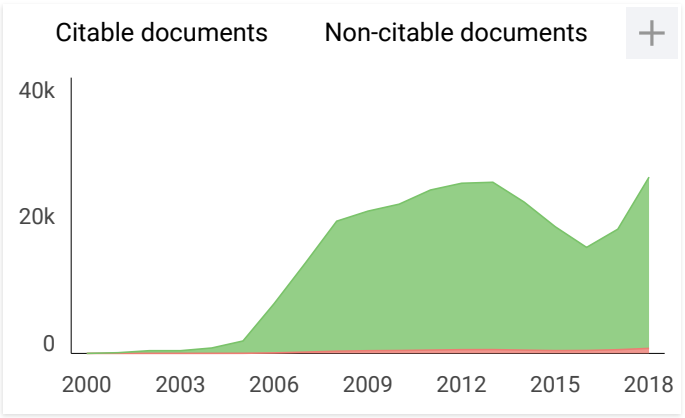

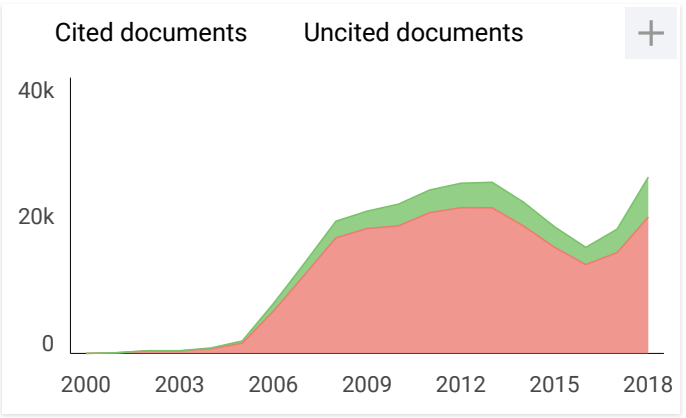

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Organic Template Free Synthesis of ZSM-5 from Calcinated Indonesian Kaolin

Hartati[1, a)], Alfa Akustia Widati[1,b)], Alfinda Novi Kristanti[1, c)], Aning Purwaningsih[1, d)], Alfiani[1, e)]

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Abstract. A pure ZSM-5 has been synthesized from calcinated kaolin without organic template. The synthesized samples were characterized using X-ray diffraction (XRD), Fourier Transform Infrared (FTIR) spectroscopy, nitrogen adsorption/desorption technique, and Transmission Electron Microscopy (TEM). The results showed that microporous ZSM-5 obtained by silica adding through three-step crystallization at 100-120-150 °C for 24 h respectively.

INTRODUCTION

ZSM-5 zeolite generally synthesized using commercial chemicals as silica and alumina sources. Tetraethylorthosilicates (TEOS) was the most chemical used as silica source [1, 2]. Other materials also used as silica sources like ludox [2], water glass [3], and SiO_2 hydrogel [4]. As alumina source, some researchers used of aluminum isoproxide (AIP) [5] aluminum sulphate ($Al_2(SO_4).18H_2O$) [3] sodium aluminates ($NaAlO_2$) [5], and other chemicals contained aluminum.

On the other hand, ZSM-5 could be synthesized using natural materials as starting materials, such as rectorite [6], rice husk ash [7], and kaolin [8,9]. The utilization of natural materials as a precursor in zeolite synthesis is more advantageous than chemicals because it is more economical.

ZSM-5 was typically synthesized using organic template as MFI structure directing agents, like tetrapropylammonium bromide, TPABr [6] or tetrapropylammonium hydroxide, TPAOH [5]. The synthesis method without MFI structure directing agents have been successfully developed and conducted [9], however it still required surfactant cethyltrimethylammonium bromide (CTAB) as mesophase agent. In the previous research, we have been successfully prepared the high crystallinity of ZSM-5 from Indonesian kaolin through treatment with the addition of sodium fluoride [10].

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Herein, we demonstrated the other technique to synthesis of ZSM-5 from metakaolin by calcination of Indonesian kaolin using step crystallization and without organic template (as a structure directing agent or as mesophase agent). The addition of tetraethylorthosilicates (TEOS) aims to complete the requirements of the mole ratio Si/Al in the composition of ZSM-5 [9]. The hydrothermal proses was conducted by controlled the temperature and time to widen the pores.

EXPERIMENTAL

Materials

The starting material of synthesis of ZSM-5 were metakaolin. The metakaolin obtained from the calcination of Indonesian (Bangka-Belitung) kaolin [9]. Synthesis was also used sodium hydroxide (NaOH) as a mineralizing agent and tetraethylorthosilicates (TEOS) as an additional silicon sources. All chemicals were purchased from Merck, analytical grade and used as received without further purification. Water used in this synthesis was distilled water.

Synthesis of ZSM-5

ZSM-5 synthesized by mixing metakaolin with solution of 2 g NaOH in 166 mL water. The mixture mixed with 54 mL TEOS and strirred for 5 h to obtaine the chemical composition of $0.25SiO_2$:$0.00625Al_2O_3$: $0.05NaOH$:$9.5H_2O$. The mixture divided by two parts, the first part aged for 24 h and other part inserted to the stainless steel autoclave. The mixture in autoclave was aged for 19 h and heated in the oven at 190^oC for 4 h. After aging, the first part poured in to autoclave and then stirred until homogeneous and heated at 100 ^oC for 24 h at the first step, and followed the second step at 120 ^oC for 24 h, and the last step at 150 ^oC for 24 h. The results washed by centrifugation process until neutral and dried at 100 ^oC for 24 h in order to obtained Z-100; Z -120, and Z-150. ZSM-5 synthesized through similar procedure with the synthesis of Z-150 but it used the addition of CTAB (Z-C) as a comparison. Sampling method conducted in each step to characterization.

Characterization

The characterization of the results with X-ray diffraction (XRD) were performed with JEOL JDX-3530 instruments using Cu Kα radiation with a step size of 0.02^o and counting time of 10 s. The samples were grinded in agate mortar before analysis. Data recorded in the 2θ range of 5-50°. Fourier Transform Infrared (FTIR) spectra of the samples measured on a Shimadzu spectrophotometer using the KBr pellet technique, in the range of 400 - 4000 cm^{-1} with a spectral resolution of 4 cm^{-1}, 45 scans, at room temperature. Nitrogen physisorption isotherms collected on a Quantachrome Nova version 10:01. The materials degassed for 5 h at 300 °C prior to analysis. Brunauer, Emmett, and Teller (BET) calculations used to determine the material surface area. Mesopores size distributions calculated using the Barrett, Joyner, and Halenda (BJH) method. Transmission Electron Microscopy (TEM) measured by JEOL, version 1.0.

RESULTS AND DISCUSSION

Metakaolin obtained form calcination of kaolin as reference [9] reported. The crystal structure of kaolin turned into an amorphous structure with calcination at 550 ^oC indicating that metakaolin had formed (Fig. 1.a and 1.b). Amorphous structure on metakaolin allows it to be used as the preparation of zeolites [11]. However, because of the XRF results on metakaolin showed SiO_2 content of 54.3% and 38.6% Al_2O_3, this research added silica of TEOS to the zeolites synthesis in order to fulfill the total mole ratio Si/Al. Metakaolin that produced from calcined kaolin showed peaks of quartz at an angle around 26.61^o as reported at reference [9] (Fig. 1.b).

Synthesis of ZSM-5 conducted through step crystallization by hidrothermal process. Diffraction patterns of the results were shown in Fig. 1.c - 1.e. Figure 1.c showed diffraction pattern of the results of synthesis by hydrothermal

process at 100^{o}C for 24 h (Z-100), by hydrothermal process at 120^{o}C for 24 h (Z-120) was shown in Fig. 1.d, and at 150^{o}C for 24 h (Z-150) was shown in Fig.1.e. Z-100 and Z-120 did not shown the formation of ZSM-5, because the diffraction pattern showed a flat curve (typical for amorphous solids). The Z-150 has demonstrated ZSM-5 because there sharp peak at 2θ about 7 - 8^{o} and about 23^{o} [1]. No evident of other peak as shown in Fig. 1.e indicates that the pure ZSM-5 has been formed. This is in accordance with the results reported by Yang et.al. [12].

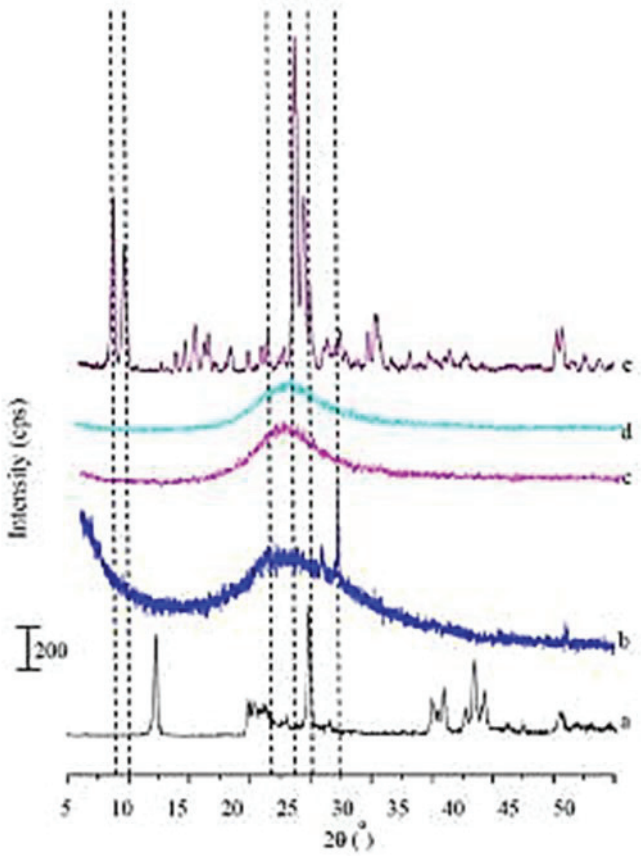

FIGURE 1. XRD pattern of samples: kaolin (a), metakaolin (b), Z-100 (c), Z-120 (d), Z-150 (e)

Figure 2 showed FTIR spectra of samples of the results. FTIR spectra of sample (Fig. 2.c) supported the XRD pattern which exposed that sample was performed ZSM-5. That was displayed by the band at around 455 ^{o}C (T – T band), 550 cm^{-1} (double five ring of MFI type zeolites), 795 cm^{-1} (external symmetric stretch), 1150 – 1050 cm^{-1} (internal asymmetric stretch), and 1224 cm^{-1} (external asymmetric stretch) [12]. While the other samples (Fig. 2.and 2.b) did not performed ZSM-5, shown by the FTIR spectra did not appear the band at around 550 cm^{-1} [12].

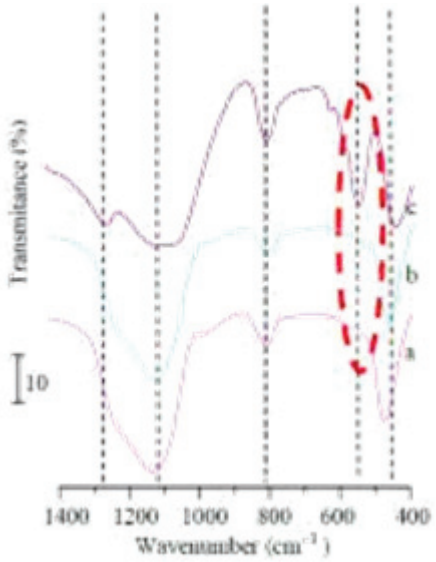

FIGURE 2. FTIR spectra of samples: Z -100 (a), Z-120 (b), Z-150 (c)

Physisorption analysis with nitrogen adsorption/desorption was carried out only on samples of Z-150 for the pure sample was formed ZSM-5 by X-ray diffraction pattern. Figure 3.a showed the nitrogen adsorption/desorption curve of Z-150. The nitrogen adsorption and desorption curve were horizontal and overlaps, which indicated that the curve as in type I, specific curve of microspores solid. This suggests that the hydrothermal gradually 100, 120 and 150^{o}C respectively for 24 hours without the addition of organic template has not been able to build up the pores of ZSM-5. The results was different from the curve of nitrogen adsorption/ desorption as shown in Fig. 3b. The data was used as a comparison, which was the result of the synthesis of ZSM-5 from metakaolin without organic template TPAOH but used template to create a mesoporous, cethyltributhylamine bromide (CTABr) (Z-C). The nitrogen adsorption/desorption curve of the Z-C contains a hysteresis loop from P/Po = 0.6 to P/Po = 1.0, indicating that the curve as in the type IV corresponding to the nitrogen filling in mesopores [1].

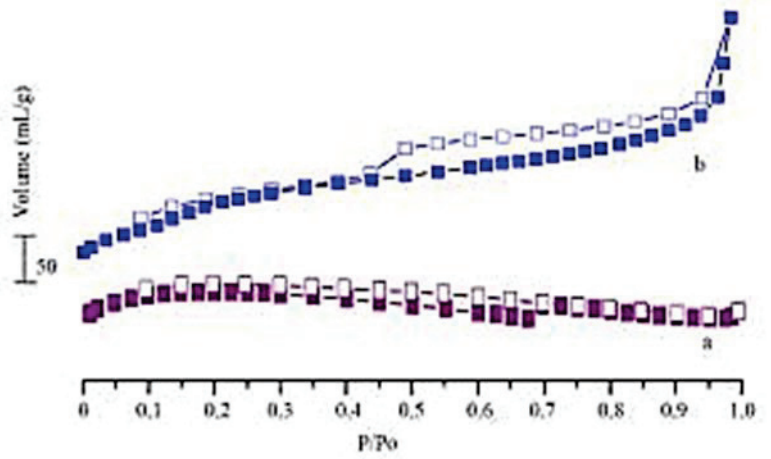

FIGURE 3. Nitrogen isotherm adsorption/desorption curve of sample: Z-150 (a) and Z-C (b)

Figure 4.a was the curve of pore size distribution of sample Z-150 which showed the curve peaks contained in diameter < 2 nm. As a comparison, the pore size of ZSM-5 which produced through metakaolin without TPAOH substance but with the addition of mesophase agent, CTABr showed the pore distribution over 2 nm (mesoporous) (Fig. 4.b). Further details listed in Table 1, which displayed the results of the analysis of the adsorption/ desorption of nitrogen for the two samples. Z-150 and Z-C showed the consecutive pore diameter of 1.691 nm and 3.836 nm. This suggests that the synthesis of ZSM-5 of metakaolin without templates organically through gradual. Hydrothermal

unable to form mesoporous and the amount of surface area mesoporous only 5.67% of the total surface area, whereas the surface area mesoporous of Z-C is 18.69% of the total area pore.

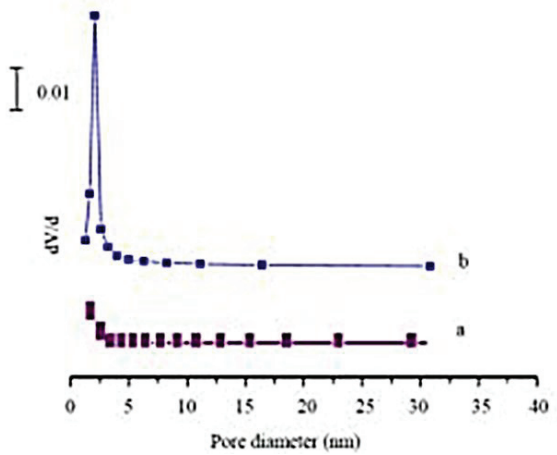

FIGURE 4. Pore size distribution of Z-150 (a) and Z-C (b)

TABLE 1. *Pore structure of ZSM-5 from metakaolin*

Sample	Mol Ratio[a] Si/Al	Surface area of mesopore[b] (m^2/g)	Surface area of micropore [c] (m^2/g)	Pore Volume (mL/g)	Pore Diameter[b] (nm)
Z-150	20	14.305	237.672	0.009	1.691
Z-C	20	67.849	295.206	0.142	3.836

a) mole ratio of synthesized condition
b) calculated by BJH method
c) calculated by BET method at $P/P_0 = 0.3$

The surface morphology of crystals analyzed by TEM which shown in Fig. 5. Figure 5 showed that the pores of the crystals on Z-150 (a) looks different from Z-C (b). The pores of the crystals in the Z-C was more obvious than the Z-150. On the contrary, the TEM image of Z-150 does not appear bright spot relative to the Z-C. The brigt spot in the TEM image can indicate of mesoporous of the solid surface [1].

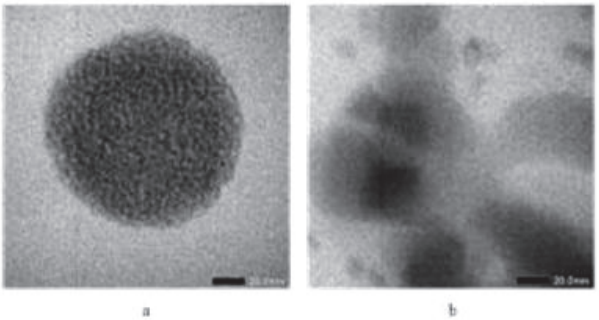

FIGURE 5. The surface morphology of Z-150 (a) and Z-C (b)

CONCLUSIONS

Pure ZSM-5 can be synthesized from Indonesia calcined kaolin with addition of silica via hydrothermal gradually without organic template at 100, 120 and 150^{o}C respectively for 24 h to produce microporous structure. The results

demonstrated the synthesis of ZSM-5 microporous, as nearly 95% of the total pore microporous surface. ZSM-5 was not performed in the synthesis by hidrothermal at 100^{o}C for 24 h (Z-100) and by hydrothermal gradualy at 100 and 120^{o}C for 24 h, respectively, (Z-120) which shown by a flat curve of diffraction pattern typical for amorphous solids.

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