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PREPARATION AND CHARACTERIZATION OF ORGANOPHOSPHATE FUNCTIONALIZED MORDENITE ZEOLITE FOR REMOVAL OF BENZEN VAPOUR IN THE AIR

Abstract. The organophosphate functionalized H-MOR materials were synthesized with a organic agent (TBP or TCP)/zeolite ratio of 10/100 (wt%) in n-hexane solvent. The obtained materials exhibited a uniform thin film structure on the surface of zeolite particles. The structure and porosity of the materials were studied by Field Electronic Scanning Emission Microscope (FESEM), X-Ray Diffraction (XRD) and Brunauer-Emmett-Teller (BET) adsorption method. Chemical bondings were characterized by Fourier Transform Infrared Spectroscopy (FT-IR). Adsorption of benzene vapor by the functionalized zeolite materials was investigated in a fixed-bed column with low benzene concentrations. The effects of factors such as empty bed contact time (EBCT), inlet concentration of benzene vapor, temperature (30°C to 50°C) and gas flow rate (0.15 L/min to 0.60 L/min) on the absorption capacity of the organophosphate functionalized zeolite were studied. The results showed that these adsorbents are suitable for application of benzene vapour treatment in the air environment.

Keywords: Mordenite zeolite, functionalized, Organophosphate, Adsorption, Benzene.

1. Introduction

Zeolites are microporous aluminosilicates which are widely used as adsorbents and catalysts in the chemical industry. In chemical engineering, zeolites, zeolite membranes are used as catalytic to remove gases and volatile organic compounds (VOCs) by combustion reactions (Bilge Yilmaz et al. [4]; Huanhoa Chen et al. [12]).

In the field of adsorption, especially adsorption of VOCs in general and benzene in particular, there

are many research results on the adsorption mechanism and the distribution of benzene molecules on some types of zeolites such as: HY (Fabien Jousse et al. [9]), MFI, CHA, BEA, STT (Coseron A.F et al. [6]), toluene adsorption on zeolite ZSM-5, MOR, and their composites such as mordenite modified with Cs, ZSM-5 zeolite membrane/PSSF (Statoshi Yamazaki, Kazuao Tsutsumi [21]; Ramiro M. Serra [20]; Huanhao Chen et al. [13]). The influence of the zeolite particle size, gas mixtures to the VOCs

adsorption process were researched and published by many authors (Ichiura, H et al. [14]; Shuai Ban [23]; Guillaume Rioland et al. [10]). When using zeolite HY as a benzene adsorbent in the air, the benzene adsorption process of zeolite HY makes up a relatively stable mixture of HY-benzene, which is still under physical adsorption; two complex forms are formed by binding of benzene to H and by the Van der Waan (vdW) interaction of benzene inside the 12-T or 4-T ring of zeolite (Shuai Ban [23]). The benzene adsorption process on zeolite HY mainly depends on the interaction of benzene with the four hydroxyl groups of four faces in a cellular unit. Typically, these H groups can be determined through IR spectrometry method (Dongjiang et al. [8]). Using the infrared spectrum for studying adsorption process of benzene in zeolites Na-Beta and H-MOR, the group of Bao-Lian Su concluded that all of the hydroxyl groups (Si-OH, Al-OH, Si-OH-Al) of the zeolites are interacting with benzene. However, only the hydroxyl groups on the 12-sided frame of the zeolite interact with benzene and the groups on the 4-sided frame are less likely to interact (Bao-Lian Su [3], Valérie Norberg 1998 and 2001). Recently, in order to increase the selective adsorption of volatile organic compounds, such as benzene, zeolite SBA-15 has been organofunctionalized with silicon mechanics, at the aim of addition of aromatic ring groups to the zeolite substrate improving the interaction between the π electrons of benzene and the $e\pi$ elements of the aromatic ring (Qin Hu et al. [19]).

Our research work has been focus on the zeolite hybrid materials with organic agents that make them have both hydrophobic and organic properties, micro/mesoporous capillary effects. These zeolite properties are able to increase selective absorption and interaction with VOCs (Nguyen et al. [18]). This paper describes the fabrication and property characterization of the organophosphate functionalized H-MOR zeolite material using TBP, TCP as organophosphate agents. Additionally, their benzene vapor adsorption ability is also studied.

Experimental

Functionalized zeolite preparation Material samples were synthesized according to the literature (Nguyen et al. [18]): 1 g of H-MOR zeolite (HS-690, Wako-Japan; $\text{SiO}_2/\text{Al}_2\text{O}_3$: 240 mol/mol; crystal size: $0.1 \times 0.5 \mu\text{m}$) was added to 50 ml of n-hexane with vigorous stirring. Next, ... g tributyl phosphate (TBP, 0.979 g/ml, Aldrich) or ... g tricresyl phosphate (TCP, 1.16–1.175 g/ml, Aldrich) taken in 50 ml of n-hexane was added dropwise to the mixture. Subsequently, the mixture was continuously ultrasonicated for 3 hours at 70°C (50W, 20 kHz) under airtight conditions to avoid evaporation of n-hexane. Finally, the products were obtained via vacuum and dried at 75°C for 1 hour. The obtained materials have been designated as $\text{CM}_{\text{PH.TBP}}$ and $\text{CM}_{\text{PH.TCP}}$ corresponding to the organic agent used TBP and TCP. The chemical bonding characteristics of $\text{CM}_{\text{PH.TBP}}$ and $\text{CM}_{\text{PH.TCP}}$ materials were evaluated by Fourier Transform Infrared Spectroscopy (FT-IR, GX-Perkin Elmer, USA), the materials structure was determined by Field Emission Scanning Electron Microscope method (FESEM, Jeol 6610LA, Japan). Nitrogen adsorption – desorption isotherms were performed at 77 K in Tristar 3000-Micromeritics equipment, USA, using static adsorption procedure. Samples were degassed at 150°C and 10^{-6} Torr for minimum 12 h prior to analysis. BET surface areas were calculated from the linear part of BET plot according to IUPAC recommendation. Pore size distributions of the samples were calculated via the conventional BJD model.

Adsorption of benzene vapor

* *Adsorption column*: 0.5 g of functionalized zeolite and 2.0 g of sand were mixed well together then packed in a 1.0 cm diameter quartz column. The length of the adsorption material packed column was 2.9 ml. In two ends of the column were blocked by layers of glass fiber. For experimental performance, the column was inserted into the thermostat room as shown in (Figure 1).

* *The research equipment:*

– Figure 1 is diagram of the equipment for organic solvent vapor adsorption investigation with

thermostat room setting temperature with vibration of $\pm 0.1^\circ\text{C}$.

Figure 1. Diagram of vapor adsorption research equipment

The equipment is operated under the principle of vacuum pressure generated by a vacuum pump. Air flow is pumped through a silicagel tube which can dry the air. The tee valve 1 and Rotameter 1 are used to regulate dried air flow which does not flow through VOCs equipment. Once the temperature of the thermal-insulated tank obtained the specific and stable value, tee valve 2 and Rotameter 2 are used to quickly drive VOCs vaporized flow to the value point of interest. The air flow carrying VOCs vaporized compounds is gone through the mixer and the column which contains 20-ml adsorption solvent subsequently. The VOCs vapor left will flow through the absorption tube and all these VOCs vapors will be grasped by activated carbons (200-g). This will ensure that exhausted gas to be emitted from the system is clean and environment-friendly.

* *Benzene concentration determination* (Abdulahma Bahrami et al. [1]; L. Zoccolillo et al. [15]): The inlet and outlet benzene vapor concentrations were determined through analysis of benzene ab-

sorbed in acetonitrile – water mixture with volume ratio of 3:1 before and after adsorption respectively by the HPLC Model HP 1100, using column C18-Zobax and DAD detector series. Dynami acetonitrile: water ratio 70:30% W; 0.005% H_3PO_4 ; 0.1% HClO_4 ; Flow rate: 1.0 ml / min; λ_{max} : 255 nm; cooling temperature in the pump system: 4°C ; Sample time: 3.

The content of adsorbed benzene vapor on the column over the time was calculated by the formula: $m_b = m_i - m_t$ (mg). In which: m_i is the amount of input benzene vapor (mg); m_t is the amount of output benzene vapor (mg) in the time t . The equilibrium adsorption capacity of the material is calculated according to the formula: $q = \frac{m_b}{m_z}$ (mg / g); In which m_z is the adsorbent mass (g).

Results and discussion

Characteristics of zeolite composite materials

The functionalized zeolite materials are composed of two types: $\text{CM}_{\text{PH.TCP}}$ and $\text{CM}_{\text{PH.TBP}}$. The formation of the functionalized zeolite is shown by the following equation:

Functionalized zeolite materials have characteristics of organophilic as they contain $\text{CH}_3\text{-C}_6\text{H}_4\text{-}$ or $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{-}$ and of hydrophilic because of having zeolite; in addition, it can be seen that the material is capillary, a type of capillary is formed by the zeolite and another type is the medium capillary due to the structure of the formed hybrid material.

The infrared spectra of $\text{CM}_{\text{PH.TBP}}$ and $\text{CM}_{\text{PH.TCP}}$ are shown in (Figure 2). The broad peak at 3662 cm^{-1} to 3490 cm^{-1} is characteristic of the vibration of the OH group of the zeolite. However, the peak intensity is weak due to the presence of physical adsorbed H_2O on the surface of zeolite, so the hydrogen bridge bonds between oxygen of water and the OH group of the zeolite surface. It was also demonstrated by the presence of peaks at 1632 cm^{-1} ($\text{CM}_{\text{PH.TBP}}$) and 1633 cm^{-1} ($\text{CM}_{\text{PH.TCP}}$) (H. Robsin [11]). The peaks at 2834 cm^{-1} and 2978 cm^{-1} characterize the C-H vibration of the CH_3 group in the tributylphosphate of $\text{CM}_{\text{PH.TBP}}$, while $\text{CM}_{\text{PH.TCP}}$ has peaks appeared at 1471 cm^{-1} and 1433 cm^{-1} , this shows that in the $\text{CM}_{\text{PH.TCP}}$ there is a characteristic vibration of aromatic cyclic structure.

In the wave numbers from 1200 cm^{-1} to 400 cm^{-1} , there appeared characteristic peaks for framework structural bonds of zeolite, both samples have the appearance of three smaller peaks around wave number 600 cm^{-1} , characterized the vibration of framework structure and distortion vibration of group

O-T-O of zeolite. For example, the occurrence of the peaks 1055 cm^{-1} and 1058 cm^{-1} which characterize the oscillation of the Si-O, Al-O bonds in the TO_4 tetrahedron of the zeolite. In addition, the wave number area less than 600 cm^{-1} , the presence of the peak at wave number 592 cm^{-1} ($\text{CM}_{\text{PH.TBP}}$) and 547 cm^{-1} ($\text{CM}_{\text{PH.TCP}}$) characterizes asymmetrical and symmetrical vibration of the five-sided ring, which is also the characteristic of distortion vibration of the double ring in the framework structure of zeolite type mordenite (Mohamed Mkhtar Mohame et al. [17]). In addition, both materials have the appearance of two peaks at wave number characterized the valence vibration of group P=O, P-O-C aliphatic for $\text{CM}_{\text{PH.TBP}}$ (1275 cm^{-1} , 1055 cm^{-1}) and P=O, P-O-C aromatic for $\text{CM}_{\text{PH.TCP}}$ (1177 cm^{-1} , 1043 cm^{-1}) (Shruti Mishra et al. [22]). These two groups indicate the presence of phosphate functional group in the structure of the functionalized zeolite.

Surface morphology of MOR, $\text{CM}_{\text{PH.TBP}}$ and $\text{CM}_{\text{PH.TCP}}$ is expressed in FE-SEM image (Figure 3). the original zeolite structure consists of almost unconnected grains with the form of rectangular blocks and the size of several μm to dozens μm . While the functionalized zeolite showed that in the grains surface likely appeared covered sticky layer. However, the surface and grains size of all three materials are almost the same as that of original zeolite. In which, the presence of P on the surface of both materials and

the carbon content of the substrate, corresponding to carbon content of 4.44% ($CM_{PH.TCP}$) and 3.80% ($CM_{PH.TBP}$), the material structure is also not sharp as with the zeolite background. But the materials

covered organic agents hopefully increase ability of VOCs separation and absorption of materials, because organic agents have the role of both absorber and conductor in interaction with VOCs.

Figure 2. Infrared spectrum of $CM_{PH.TBP}$ (a) and $CM_{PH.TCP}$ (b)

Figure 3. The FE-SEM image of sample MOR; CM_{PH.TBP} and CM_{PH.TCP}

For better understanding the changes in the structure of the functionalized zeolite materials, the porosity of the materials was studied via the

nitrogen isothermal adsorption-desorption method. The N₂ adsorption-desorption isotherms of all sample are presented in (Figure 4).

Figure 4. Diagram of the nitrogen isothermal adsorption-desorption line and capillary size distribution of CM_{PH.TBP} and CM_{PH.TCP}

Study on the surface properties of functionalized zeolite materials, based on the BET method shows that the nitrogen isothermal adsorption-desorption line of all three samples was of type IV and has hysteresis loop type H2 (as sorted by IUPAC). The belong to materials have medium capillary with hollow both ends cylinders, and corresponding to the Barret-Joyner-Halenda (BJH) hole size distribution.

Figure 4 also shows that desorption line of the samples were relatively smooth, in which the sample CM_{PH.TBP} has the nitrogen isothermal adsorption-

desorption line started condensation in the relative pressure P/P_0 about 0.5–1.0 demonstrates that the material has a relatively large capillary diameter.

While CM_{PH.TCP} sample of late loop is not closed loop at low pressure of P/P_0 , indicating the presence of organic agents in the sample alters TCP ring laticy, as well as changes in the distribution of the capillary size of the sample than the sample of CM_{PH.TBP} (Dongjiang Yang et al. [8]).

In which, the specific surface area and average capillary diameter of sample MOR; CM_{PH.TBP}

and $CM_{PH.TCP}$ are 474.47 m²/g; 329.65 m²/g; 385.40 m²/g and 2.4614 nm; 2.6479 nm and 2.5878 nm respectively. Based on the above results, the $CM_{PH.TCP}$ material has the smallest surface area, with the largest average capillary size, indicating a greater percentage of capillary size than the percentage of large capillary size of both MOR

and $CM_{PH.TBP}$ material. This shows that the functionalized zeolite material based on MOR zeolite and TCP organic matter, TCP acts as a framework for the formation of large capillary sizes and also as a VOCs vapor conductor when applied in later processing.

Figure 5. TG and DTA plots of $CM_{PH.TCP}$ [1], $CM_{PH.TBP}$ ([2]) and MOR ([3])

TG and DTA curves (figure 5) show that, when rising room temperature to 300° C, the sample mass decreases rapidly. In both samples, the effect of heat recovery was 199.0° C ($CM_{PH.TBP}$) and 304.5° C ($CM_{PH.TCP}$). This effect may be the escape of organic solvent or water vapor in the capillary structure of the studied materials. Continuously increasing the temperature to more than 300° C, the studied materials have reduced mass, but the $CM_{PH.TCP}$ and $CM_{PH.TBP}$ have more mass reduction than the MOR.

From the results of the study, there are differences in the thermal effect at these two temperatures, due to the different physicochemical properties of the two compounds. In particular, the TCP agent has a molecular weight of 368.37

g/mol and a flammability temperature of 220° C. The TBP agent has a molar mass of 266.32 g / mol and is particularly flammable at 146° C is much lower than the TCP counterpart, so the thermal effect of the material $PH_{PH.TCP}$ occurs at a higher temperature than the thermal effect occurs with the material $CY_{PH.TBP}$.

Noteworthy, the particle size of the material is almost the same as that of the zeolite substrate, which shows that the film on the surface of the material is rather thin, with the organic matter deposited on the substrate surface to increase its ability to adsorb subtraction and gas separation of the material, as organic agents both act as carriers and interact with VOCs (Qin HU et al. [19]; Mohamad et al. [16]).

Adsorbability of benzene vapor on zeolite composite materials

Benzene vapor adsorption by the functionalized zeolite materials was conducted with the gas flow rate from 0.15 L/min to 0.6 L/min at 40 °C, the inlet benzene concentration of 21.8 ppmv and the bed volume of 2.9 mL.

Empty bed contact time is one of the important parameters of an adsorption system. Assuming the

flow is ideal, the empty bed contact time is calculated by the following formula (Chaiwat et al. [5]; William et al. [24])

$$EBCT = \frac{V}{f}$$

where $EBCT$ is empty bed contact time, s

V – is empty bed volume of the column, L.

f – is the gas flow rate through the column, L/s.

The results are given in table 1 and (Figure 6).

Table 1. – Influence of gas flow rate to the adsorption capacity of $CM_{PH.TBP}$, $CM_{PH.TCP}$

The gas flow rate (L/min)	Contact time (s)	Adsorption capacity of $CM_{PH.TBP}$ (mg/g)	Adsorption capacity of $CM_{PH.TCP}$ (mg/g)
0.15	1.16	1.0607	1.5014
0.30	0.58	1.1005	1.4832
0.45	0.32	0.9973	1.4075
0.60	0.29	0.9012	1.3297

As results showed in (table 1), with the same inlet benzene concentrations, but with lower contact time (about 0.29 s), the adsorption capacity began decrease. It demonstrates the rate of gas flow significantly affects to the adsorption capacity of the materials. Particularly, when the contact

time is too short, it shall not guarantee that gas molecules can diffuse through the absorbed material surface. From the above review, we conducted the survey of benzene vapor adsorption process of the materials in the two flow rate 0.15, 0.3 and 0.45 L/min.

Figure 6. Breakthrough curve of benzene vapor through zeolite composite column at 40 °C

The results also showed that at the flow rate of 0.15 and 0.45 L/min, the adsorption capacity of the materials varied not so much. However, adsorption breakthrough curve is different. The breakthrough

curve at a flow rate of 0.45 L/min is more sloping than those of 0.15 L/min and 0.30 L/min. However, the difference of adsorption capacity appeared by the different organophosphorous component in the

material. The composite material with tributylphosphate showed higher adsorption capacity than those with tricresylphosphate.

The investigation was carried out at 40 °C and 50 °C; the initial benzene concentration of 21.8

ppmv and 32.6 ppmv; air flow rate 0.3 L/min; the adsorption fractions were measured after every 5 min. The cumulative curves of benzene vapor on the functionalized zeolite materials are given in (Figure 7).

Figure 7. Cumulative curves of benzene vapor on the zeolite composites at 40 °C and 50 °C

The results showed that, increasing of temperature from 40 °C to 50 °C, the adsorption capacity of CM_{PH.TBP} and CM_{PH.TCP} increased, and after maximum 60 min contacted the adsorption process of all materials reached equilibrium state. The adsorption capacity of CM_{PH.TBP} reached 1.0917 mg/g and 1.8031 mg/g, and those of CM_{PH.TCP} were 1.5014 mg/g and 2.9725 mg/g respectively. This phenomenon seems unaccordant to the adsorption kinetics (temperature increased, the adsorption capacity decreased). In this case, it can be explained by better diffusion of benzene through organophosphorous layer to zeolite core under high temperature and holed in zeolite structure. But actually, the threshold of higher temperature was not determined yet. The adsorption capacity of benzene vapor on CM_{PH.TCP} was better than that on CM_{PH.TBP} and original zeolite. The reason can be explained by that, the IR study showed that the CM_{PH.TCP} has interaction of benzene ring in the TCP agent with benzene vapor, so the dynamic equilibrium adsorption capacity of this material was greater than that of CM_{PH.TBP}. According to Qin Hu et al. 2009, the presence of aromatic cyclic

agent in the material, its adsorption ability changed, and this was explained by the interaction of electron- π of benzene or phenyl when studied adsorption of benzene on different materials.

Conclusion

This article presents new experimental results on functionalized zeolite material. This material is prepared from zeolite MOR with tributylphosphate (CM_{PH.TBP}) and tricresylphosphate (CM_{PH.TCP}). As a result, two materials of CM_{PH.YBP} and CM_{PH.TCP} having large specific surface area of 329.65 m²/g and 385.40 m²/g with current pore data around 2.6479 nm to 2.5878 nm respectively were obtained. The BET surface area, TG, FT-IR spectra of the functionalized zeolite showed strong interaction between zeolite base and organophosphate substances.

Adsorption of benzene vapor in fixed-bed columns on functionalized zeolite materials, in quite low concentration from 20 ppmv to 40 ppmv and the adsorption time of 0.29 s to 1.16 s, the equilibrium adsorption capacity of CM_{PH.TBP} reached 1.0917 mg/g and 1.8031 mg/g, and those of CM_{PH.TCP}

were 1.5014mg/g and 2.9725 mg/g corresponding with 40 °C and 50 °C in the adsorption conditions of gas flow rate of 0.3 l/min and input benzene concentration of 21.8 ppmv and 32.6 ppmv.

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