SYNTHESIS OF MESOPOROUS ALUMINOPHOSPHATES AS
POTENTIAL CATALYST IN THE UPGRADING PETROLEUM
FEEDSTOCKS

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By
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Synthesis of Mesoporous Aluminophosphates as Potential Catalyst
in the Upgrading Petroleum Feedstocks

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II ABSTRACT

This project focuses on the synthesis of mesoporous aluminophosphate (AlPO) catalysts for application in the acid catalyzed conversion of large petroleum feedstock compounds to useful middle distillates and naphtha transportation fuels. Summarized herein, is our research progress for September 1, 2000 to August 31, 2001. The liquid crystalline templating approach was used for the synthesis of mesoporous aluminophosphates. A systematic study of the influence of synthesis variables (gel composition, time and temperature) was carried out. In general, syntheses were conducted by combining aluminum and phosphorus precursors, cetyltrimethylammonium chloride (CTACl) as surfactant, tetramethylammonium hydroxide (TMAOH) and water with typical molar composition of the synthesis mixture as follows: $x\text{Al}_2\text{O}_3:\text{P}_2\text{O}_5:0.5\text{CTACl}:2.6\text{TMAOH}:35\text{H}_2\text{O}$, where $x=0.29\text{--}2.34$. A variety of hexagonal and lamellar ALPO_4 with pores size distribution in the $20\text{\AA} - 40\text{\AA}$ range were prepared in the presence of long chain CTACl surfactant. The products depended on the molar composition of the synthesis mixture. The aluminum source had significant influence on the products obtained. Products formed using pseudoboehmite alumina (catapal B), were more stable than those formed with aluminum isopropoxide. Aluminum hydroxide gave thermally unstable products. The influence of these and other synthesis variables is presented.

1.0 INTRODUCTION

This project focuses on the synthesis of mesoporous aluminophosphate (AIPO) catalysts for application in the acid catalyzed conversion of large petroleum feedstock compounds to useful middle distillates and naphtha transportation fuels. Our approach utilizes large surfactant micelles as the templating agents around which "silicon and transition-metals-containing mesoporous catalysts with pores size in the 2-10 nm range are synthesized. The rationale is based on the knowledge that individual surfactant molecules are known to aggregate when they are present in solution at or above certain concentrations - the critical micellar concentration (CMC). In the case of micelle-templated mesoporous materials, the individual surfactant monomer contained in each micelle, is extended across the cross-section of the pore. The length of the surfactant molecule defines the pore size of the resulting material.

This report summarizes our research progress for September 1, 2000 to August 31, 2001, and highlights the influence of some reaction conditions on the product obtained.

2.0 EXPERIMENTAL

In general, syntheses were conducted by combining aluminum and phosphorus precursors, cetyltrimethylammonium chloride (CTACl) as surfactant, tertamethylammonium hydroxide (TMAOH) and water. The reactants were as follows: CTACl (25 % solution in water, TMAOH (25 % solution), H_3PO_4 (85 %), and water. In a typical synthetic series, an accurately weighed amount of aluminum precursor was slowly added to a solution of 4.2 g of phosphoric acid in 15 g of water, with vigorous stirring. The mixture was added to a solution of 11.6 g of CTACl in 100 g water with stirring. After 0.5 hr, 17.3 g of TMAOH (25 % solution) was slowly added dropwise into the above mixture. The molar composition of the resulting synthesis mixtures were: $x\text{Al}_2\text{O}_3$, P_2O_5 : $y\text{CTACl}$: $z\text{TMAOH}$: $w\text{H}_2\text{O}$, where x, y, z and w were systematically varied. The reaction mixtures were stirred for various times and temperatures. The solid products were recovered, washed and air-dried at 70°C.

Powdered X-ray diffraction measurements were made on a Phillips X-Pert X-Ray Diffractometer using Cu-K α radiation ($\lambda = 0.15418$ nm) between 1 and 10 two theta. Nitrogen adsorption isotherms and BET surface area measurements were obtained using a Micromeritics Gemini 2310 under liquid nitrogen temperature. The samples were preheated at 200°C in nitrogen for 3 hr, then evacuated prior to adsorption measurement. Pore size distribution was calculated using the Barrett-Joyner-Halenda (BJH) model. Thermogravimetric analysis was performed using a TA Instrument model SD2960 Thermal Analyzer.

3.0 RESULTS AND DISCUSSION

3.1 Synthesis of mesoporous aluminophosphates at various Al/P ratios and using aluminum hydroxide as source of Al

Our initial approach was based on the use of aluminum hydroxide as the aluminum source. Aluminum hydroxide, with its ease of hydrolysis, is widely used in the synthesis of microporous aluminophosphates but had limited success in the synthesis of the mesoporous aluminophosphate counterparts¹. Whereas, in the microporous aluminophosphates an Al/P of 1:1 is optimum for their synthesis, it is known that for the mesoporous counterparts, variation in the Al/P ratio can have significant effects on the type and quality of the product obtained. We, therefore, decided to investigate the effect of Al/P in our synthesis. The molar composition of the synthesis mixtures were: $x\text{Al}_2\text{O}_3, \text{P}_2\text{O}_5:0.5\text{CTACl}:2.6\text{TMAOH}:35/\text{H}_2\text{O}$, where $x = 0.29 - 2.34$

Figures 1 shows the X-ray diffraction patterns of products obtained as the Al/P ratio was varied from 0.29 to 0.34. At the low Al/P ratio (0.29) sharp diffraction peaks corresponding to a lamellar phase^{2,3} were observed, with the first peak occurring at 2θ of 2.51° ($d = 35$ Å). As the aluminum content was increased to a Al/P ratio of 1.25, the diffraction pattern changed to peaks corresponding to an hexagonal phase, with the first peak appearing at 2θ of 2.18° ($d = 40.5$ Å) and a second at 6.02° ($d = 14.7$ Å). Diffraction peaks were broader than the lamellar phase and the first peak position shifted to lower 2θ values (higher d spacing) as the aluminum content increased. The sharpest peaks corresponding to the hexagonal phase were observed in the Al/P ratio range of 0.47 -1.25. Beyond Al/P of 1.25, a very small and diffused peak was observed at a 2θ of 1.77° , possibly corresponding to the formation of a very small amount of poorly crystalline phase.

Figure 1. XRD of mesoporous aluminophosphates synthesized under various Al/P ratios.

Figure 2 shows the changes in the pH of the reaction mixture as a result of the variation in the Al/P ratio. The most crystalline hexagonal products were obtained in the pH range of 8 to 9.5 corresponding to Al/P of 0.47 to 1.25. Whether it was the variation in the Al content or pH values that resulted in differences in the products (lamellar vs hexagonal) was not clear from this experiment, but may be obvious in later experiments.

Figure 2. Variation of pH in reaction mixture for aluminophosphate synthesized at various Al/P ratios.

The development of porosity in the products can be inferred from desorption of occluded species using thermogravimetric analysis. Figure 3 shows thermograms of products obtained at various Al/P ratios. The corresponding weight loss events for these products are presented in Table 1. Desorption events in the regions of 200°C, 350°C, and 500°C for sample with Al/P ratio of 0.59 for example, are ascribed to water loss, decomposition of CTA, and TMA respectively. Samples with Al/P ratio ≥ 1.25 showed relatively small loss of organic species, compared with samples synthesized at lower Al/P ratios. Lower Al/P ratio samples showed desorption of CTA corresponding to approximately 40-55 % of sample wt.

Figure 3. Thermograms of mesoporous aluminophosphates synthesized under various Al/P.

Table 1. Weight loss events for mesoporous aluminophosphates

Al/P 0.29		Al/P = 0.33		Al/P= 0.47		Al/P=0.58		Al/P = 1.25		Al/P=2.34	
Temp (C)	Wt. Loss %	Temp (C)	Wt. Loss %	Temp (C)	Wt. Loss %	Temp (C)	Wt. Loss %	Temp (C)	Wt. Loss %	Temp (C)	Wt. Loss %
25 - 186	5.5	53-187	8.6	54-190	10	66-201	9.8	97	15	53	6.1
186 - 353	52	187-338	55	190-337	46	201-351	41	210-336	27	122-341	33
353 - 500	9.8	338-500	3.6	337-500	4.6	351-500	9.2	336	13	341	17
Total Wt Loss	68		67		61		60		54		56

Figure 4. XRD of aluminophosphates synthesized under various TMAOH/P₂O₅ ratios with aluminum hydroxide as Al source.

The creation of mesopores is generally accomplished by calcination of the "as synthesized" material. Attempts to remove the organics from the samples by calcination between 300°C and 500°C under nitrogen or nitrogen/air resulted in structural collapse. This was determined from the loss of peaks in the diffractograms and very low surface areas/pore volumes. Using solvent extraction (sodium acetate/ethanol) as an alternative organics extraction strategy resulted in a similar structural collapse. Samples with Al/P ratio of 1.24 yielded surface areas of 400 m²/g. High Resolution Transmission Electron Microcopy analysis is pending at this time, hence pore architecture of the samples is not yet determined.

It was determined that mesoporous materials formed under the conditions detailed above were of low thermal stability, and therefore, other synthesis variables should be investigated to study their influence on the thermal stability of the materials. As a result, the next step was to investigate other synthesis variables which could increase the crystallinity, and therefore, structural stability of the resulting mesoporous ALPOs.

3.1.1 Effect of TMAOH Concentration

In this experiment, the amount of TMAOH added to the synthesis mixture was varied in an effort to impact the rate of hydrolysis of the aluminum hydroxide. Synthesis mixtures containing varying amounts of TMAOH were prepared for three different Al/P ratio with the following molar compositions.

where $w = 0 - 3.67$.

The reaction mixtures were prepared under otherwise similar conditions described in Section 2.1.

Figure 4 shows the diffractograms for products obtained from variation in TMAOH content of the synthesis mixture with molar composition of $0.59\text{Al}_2\text{O}_3:\text{P}_2\text{O}_5:0.5\text{CTACl}:w\text{TMAOH}:35/\text{H}_2\text{O}$ and after 24 hrs of reaction time at room temperature. Figure 5 shows the corresponding changes

on pH of the reaction mixture. With no TMA or a low TMA/P₂O₅ ratio of 1.06, (pH 5.22) an amorphous product was obtained. As the TMA/P₂O₅ ratio increased to 1.47 (pH 6.51) the resulting product showed diffraction peaks of relatively low intensities. The peaks were assigned to the presence of both lamellar and hexagonal phases. TMA/P₂O₅ in the range of 2.20 - 3.67 (pH 7.95-10.37) yielded products with peaks of higher intensities corresponding to hexagonal phase only but there was no obvious trends in the peak characteristics. The product obtained by extending the reaction time from 24 hr to 72 hr, showed a similar diffraction pattern.

Products obtained from heating the reaction mixtures to 110°C under otherwise similar conditions, gave similar results (Figure 4), except that a lamellar phase was observed at a TMA/P₂O₅ ratio of 2.18. A hexagonal phase was observed at TMA/P₂O₅ ratio of 3.46 (pH 9.51), but at a higher TMA/P₂O₅ ratio of 3.67 (pH = 3.67) or extended reaction time (72 hr) the product was of lower crystallinity.

Figure 5. Variation of synthesis pH vs TMA/P₂O₅ mole ratio.

Similar trends were observed for products formed from reaction mixtures of molar compositions of higher Al/P ratios (*1.25Al₂O₃:P₂O₅* and *1.50Al₂O₃:P₂O₅*). However, less crystalline phases were consistently observed in products formed from mixtures with Al/P ratio of 1.50 over the TMA/P₂O₅ range studied.

3.1.2 Effect of Mixing time

The pore volume and pore size distribution of the aluminophosphates are expected to depend on the mixing time of aluminum and phosphorus reactants prior to the addition of the other reagents. This may be related to the influence of acid hydrolysis (time) of the aluminum hydroxide on the crystallinity and stability of the resulting products. Experiments were, therefore, conducted to vary the mixing time of aluminum hydroxide and phosphoric acid mixture from 2 to 6 hr prior to the addition of CTACl and TMAOH for two molar compositions; - $x\text{Al}_2\text{O}_3:\text{P}_2\text{O}_5:0.5\text{CTACl}:2.6\text{TMAOH}:35/\text{H}_2\text{O}$, where x was 0.59 and 1.25.

Figure 6. Adsorption isotherms of mesoporous aluminophosphates synthesized with aluminum hydroxide as Al source.

For materials synthesized with Al/P ratios of 1.25 (Figure 6), and 0.59 (Figure 7), it was observed that the longer the mixing time (4 hr and 6 hr), the larger the pore volume of the product. Also, the higher the Al content in the synthesis mixture, the larger the pore volume of the product.

In all cases the pH of the final synthesis mixture ranged from 8.11 to 9.98. Varying the mixing time showed no appreciable trend in the XRD of the product. All products obtained at low Al/P

ratio ($x = 0.59$) show a fairly well defined characteristic first diffraction line at 2θ of 2° . With calcination at 400°C , the major peak becomes broader, shifting to higher diffraction angles and loses some intensity. Products obtained at high Al/P ratio ($x = 1.25$) showed less defined x-ray peaks, but the shift observed in the major characteristic peak position was smaller than the case described above. In this case, the pH of the final reaction mixture range from 9.08 to 8.11 after addition of the final reactant to 10.8 to 7.48 at the end of the reaction time.

Porosity measurements showed that samples synthesized at high Al/P ratio had larger surface areas and pore volumes. Nitrogen isotherms of calcined samples shown in Figure 6 were intermediate between Types I and IV. Sample with low Al/P ratio (0.59) stirred for 2 hr, gave no isotherm. Sample with Al/P ratio of 1.25 gave consistently higher total pores volume compared with samples of Al/P ratio of 0.59.

Figure 7. Pore volume and pore size distribution of aluminophosphates synthesized with various mixing times and Al/P ratio of 1.25.

The samples showed broad pore size distribution which peaked at about 12 Å , and hence in the microporous domain (Figure 7).

3.1.3 Effect of synthesis time

Using a mixing time of 2 hr after prior to the addition of TMAOH, synthesis was carried out at ambient temperature using four synthesis mixtures, each with molar composition of $0.59\text{Al}_2\text{O}_3:\text{P}_2\text{O}_5:0.5\text{CTACl}:2.6\text{TMAOH}:35/\text{H}_2\text{O}$. Mixtures were allowed to react under vigorous stirring conditions for various times up to 48 hrs. Intermediate products during the course of the reaction were examined. The degree of their crystallinity, represented as intensity of the (100) XRD reflection, increased during the course of the reaction (Figure 9). The hydrothermal reactions were practically complete after 24 hrs.

Figure 8: Intensity of the 001 XRD reflection vs synthesis time.

Calcination of all samples in nitrogen and nitrogen/air between 400 C and 500 C resulted in some loss of intensities (complete loss for 5hr and 10 hr samples) and a broadening and shift of the main peak to higher 2θ angles.

3.1.4 Effect of CTACl concentration

The amount of CTACl was varied in an effort to vary micellar concentration of the surfactant with molar composition $0.59\text{Al}_2\text{O}_3\text{:P}_2\text{O}_5\text{:}x\text{CTACl:}3.6\text{TMAOH:}35\text{H}_2\text{O}$, where $x = 0.25 - 1.00$. At low CTA/ Al_2O_3 ratio, a poorly defined single (100) reflection was observed, which became more defined as the CTA/ Al_2O_3 concentration ratio was increased from 0.34 to 0.73. At CTA/ Al_2O_3 of 1.00, lamellar phase was observed. The type and crystallinity of the product was independent of temperature between 25°C and 110°C.

3.2 Effect of aluminum source

Though XRD and thermal analysis indicated the formation of mesoporous materials using aluminum hydroxide as Al source, the thermal instability of the resulting materials made it unable to calcine the materials and hence no pore size distribution could be determined. We thought that the fast hydrolysis of the hydroxide may have resulted in too many aluminate species available for nucleation of the materials and hence not enough species remaining for subsequent pore wall thickening, which would render the materials resistant to thermal collapse.⁴ It was thought that slowing the rate of generation of hydrolysis species would slow the nucleation process and more aluminate species would be available for wall thickening. Aluminum isopropoxide (Aldrich) and pseudoboehmite (catapal B, Vista) were therefore selected, as they have slower rates of hydrolysis in comparison to the hydroxide.

3.2.1 Synthesis of mesoporous aluminophosphates at various Al/P ratios and using aluminum isopropoxide as source of Al

In a typical synthetic series, an accurately weighed amount of aluminum isopropoxide was slowly added to a solution of 4.2 g of phosphoric acid in 15 g of water with vigorous stirring. The mixture was stirred added to a solution of 11.6 g of CTACl (25 % solution)) in 100 g water with stirring. After 0.5 h, 17.3 g of TMAOH (25 % solution) was slowly added, dropwise, into the above mixture.

Figure 9: Adsorption isotherms for aluminophosphates vs Al/P ratio.

The reaction mixture was stirred for 72 hrs at room temperature. The solid product was recovered and air-dried at 70°C. The molar composition of the resulting synthesis mixtures were as follows: $x\text{Al}_2\text{O}_3, \text{P}_2\text{O}_5:0.5\text{CTACl}:2.6\text{TMAOH}:35/\text{H}_2\text{O}$, where $x = 0.29\text{--}2.34$.

Figure 10: Pore size distribution of aluminophosphates at different Al/P ratio.

Product obtained at Al/P ratio of 1.17 yielded a Type I isotherm with inflection at P/P_0 of 0.3; whereas product with Al/P of 0.59 gave a Type I-IV combination (Figure 9). The high Al/P

Figure 11. Adsorption isotherms of aluminophosphates vs various mixing time (Al/P=1.17).

Figure 12: Pore size distribution of aluminophosphates vs mixing times (Al/P = 1.17).

ratio product shows a broad pore size distribution that is centered about 20 Å, whereas low Al/P ratio product gave unresolved pore size distributions (Figure 10). For reaction mixtures with Al/P ratio of 1.17, longer mixing times gave products with higher absorption volumes, but with broad pore size distribution (Figures 11 and 12). For reaction mixtures with lower aluminum content (Al/P ratio of 0.59), longer mixing times gave products with lower absorption volumes, but with broad pore size distribution (Figures 13 and 14).

Figure 13: Adsorption isotherms of aluminophosphates formed using various mixing times (Al/P=0.59).

Figure 14: Pore size distribution of aluminophosphate formed at different mixing time (Al/P = 0.59).

3.2.2 Synthesis of mesoporous aluminophosphates at various Al/P ratios and using catapal B alumina as source of Al

Aluminophosphate synthesis was conducted using procedure outline in Section 2.1.1, except that

Figure 15. Adsorption isotherms of aluminophosphates formed using catapal B.

catapal B alumina was used in place of aluminum hydroxide. The molar composition of the resulting synthesis mixtures were as follows: $x\text{Al}_2\text{O}_3:\text{P}_2\text{O}_5:0.5\text{CTACl}:2.6\text{TMAOH}:35/\text{H}_2\text{O}$,

Figure 16: Pore size distribution of aluminophosphates formed at various Al/P ratio.

where $x = 0.59-2.35$. Product formed with catapal B alumina, shows in general, Type 1 isotherms, with relatively sharp inflections around P/P_0 of 0.4. Sharpest inflections were observed for products of Al/P ratios of 1.18 and 2.35. Pore size distributions for these products were in the region 40Å and 25Å respectively (Figure 16). Powdered XRD patterns of the products are awaiting pending analysis.

3.2.2.1 Effect of water on synthesis with catapal B alumina

The amount of water was varied in an effort to vary the rate of hydrolysis of the catapal B. Synthesis mixtures containing varying amounts of water were prepared with a molar composition of $1.18\text{Al}_2\text{O}_3:\text{P}_2\text{O}_5:0.50\text{CTACl}:2.84-3.22\text{TMAOH}:z\text{H}_2\text{O}$, where $z = 87-700$.

Figures 17 and 18 shows that $\text{H}_2\text{O}/\text{P}$ ratio of 350 gave product with pore size around 22 Å, and $\text{H}_2\text{O}/\text{P}$ between 43 and 175, gave product with pore distribution between 31 to 38 Å.

Figure 17. Adsorption isotherms of aluminophosphates vs water content of synthesis mixture.

Figure 18: Pore size distribution of aluminophosphate vs water content of synthesis mixture.

4.0 CONCLUSIONS

Mesoporous aluminophosphate can be synthesized by combining aluminum and phosphorus precursors, cetyltrimethylammonium chloride (CTACl) as surfactant, tetramethylammonium hydroxide (TMAOH) and water with typical molar composition of the synthesis mixture as follows: $x\text{Al}_2\text{O}_3:\text{P}_2\text{O}_5:y\text{CTACl}:z\text{TMAOH}:w\text{H}_2\text{O}$. A variety of hexagonal and lamellar ALPO_4 with pores size distribution in the $20\text{\AA} - 40\text{\AA}$ range were prepared in the presence of long chain CTACl surfactant. The product depends on the molar composition of the reaction mixture. Among the synthesis variables investigated, the aluminum source had the most significant influence on the products obtained. Products formed using pseudoboehmite alumina (catapal B), were more stable than those formed with aluminum isopropoxide. Aluminum hydroxide gave thermally unstable products.

5.0 CHALLENGES

The most significant setback experienced in the lack of access to a High Resolution Microscope. This prevented the direct observation of pore size architecture in the materials synthesized.

6.0 FUTURE WORK

Work in the immediate future will focus on exploring other reaction variables to synthesize mesoporous aluminophosphates with sharper pore size distributions and higher thermal stabilities. Subsequently, we will synthesize mesoporous aluminophosphates containing other metals such as, Mg, Ni, Co and V in the mesoporous framework. The incorporation of these metals will simultaneously introduce acid functionality in the materials. The products will be evaluated as catalysts to upgrade petroleum feedstocks.

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