



Structural Study of Gamma Alumina (γ -Al₂O₃) and β Co-Crystalline Zeolite

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ABSTRACT

The effect of preparation by ion exchange method and impregnation aqueous Hexachloroplatine acid (40 wt. % Pt) on structural changes were studied. Two synthetic zeolites of different compositions were prepared by ion exchange using (NH₄CL) and calcinations at 350°C for 2hr. Then subsequently loaded with 0.5 wt. % Pt by impregnations with aqueous hexachloroplatine acid (H₂PtCl₆.6H₂O) and calcinations and reduced with hydrogen. The compositions analyzed before preparation by X-ray Florence (XRF) and X-ray Diffraction (XRD) and then analyzed after preparation via Scanning Electronic Microscopy (SEM). The morphology and average crystallite size of γ -Al₂O₃ was 7 μ m, while for β Co-Crystalline Zeolite was 8.5 μ m. Platinum particles were found to be homogeneously distributed and the crystallite faujasite size range (5-10) μ m. When analyzed via Energy Dispersive X-ray (EDAX) they agree well with (XRD) and zeolite composition, and analyzed via Brunauer Emmett Teller (BET) the results of Surface Area m²/mg giving the highest values equivalent 288.86m²/mg and 427.98m²/mg for Pt/H- γ -Al₂O₃ and Pt/ H- β Co-Crystalline Zeolite respectively and Pore Volume cm³/gm equivalent 0.3199cm³/gm and 0.6603cm³/gm respectively.

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INTRODUCTION

Metal oxides represent one of the most important and widely employed

categories of solid catalysts, either as active phases or as supports. Metal oxides are utilized both for their acid-

base and redox properties and constitute the largest family of catalysts in heterogeneous catalysis (Manoj *et al.*, 2012). A typical catalyst comprises one or more catalytically active component supported on a catalyst support. Typically, the catalytically active components are metals and/or metal-containing compounds. The support materials are generally high surface area materials with specific pore volumes and distribution (Lovasic *et al.*, 1990; Raouf, 1994; Novaro *et al.*, 2000; Ramze *et al.*, 2008). Ion exchange is normally mediated by a liquid phase. In special cases, however, direct exchange between solids has also been observed: In mixtures of metal halides with dehydrated ammonium or hydrogen zeolites, ion exchange proceeded at temperatures typically above 570 K and resulted in the evolution of ammonium and/or hydrogen halide and in the formation of the corresponding metal-cation form of the zeolite concerned (Gabriella *et al.*, 1989). Impregnation of pore

supported catalyst is achieved by filling the pores of support with solution of active species of metal salt from which solvent is evaporated. The concentration of the metal content can be increased by successive impregnation with intermediate precipitation and thermal activation to insolubilize the supported species (Jens, 1997; Shuguang *et al.*, 2000). Characterization of the catalyst is a predominate step in every catalyst study and at every stage of the catalyst development. Critical parameters are measured not only to check the effectiveness of each operation, but also to provide specification for future products. Characterization might be studied or controlled in terms of supported properties, metal dispersion and location and surface morphology (Thomas, 2004).

EXPERIMENTAL METHODS

The Preparation of Pt/H- γ Al₂O₃ and Pt/H- β Co-Crystalline Zeolite is shown in Figure (1). After preparation by ion exchanging and impregnation.

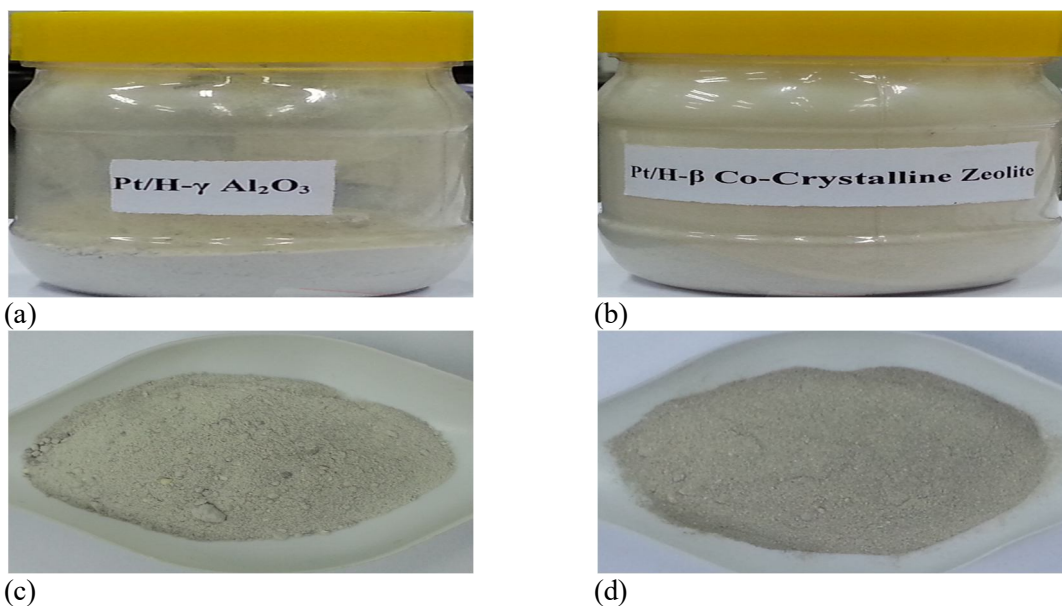


Figure 1: a and c: Pt/H- γ Al₂O₃ and b and d: Pt/H- β Co-Crystalline Zeolite

Preparation of H- γ -Al₂O₃ and H- β Co-Crystalline Zeolite by Ion Exchange:

This was prepared by ion exchanging of the parent H- γ -Al₂O₃ and H- β Co-Crystalline Zeolite with (3N) ammonium chloride solution. Thus, 32.094gm of ammonium chloride in 200ml distilled water were added to 40gm of zeolite with stirring for 1hr. At 50°C. The batch of salt was left in the solution for 72hr. At 25°C. The exchanged ammonium salt was then filtered off, washed with deionized water to be free of chloride ions and dried at 110°C overnight. The dried samples were then calcined at 500°C for 7hr. In presence of O₂.

Impregnation of H- γ Al₂O₃ and H- β Co-Crystalline Zeolite with platinum:

The hydrogen exchanged zeolites were loaded with 0.5 wt. % Pt by impregnation with an aqueous solution of Hexachloroplatinic acid (H₂PtCl₆.6H₂O). The platinum content of the catalyst was the calculation of the weight of the support and the amount of the metal in impregnation solution. Thus, 0.5gm of Hexachloroplatinic acid (40 wt. % Pt) were dissolved in 50ml of distilled water. Then the solution was added for 40gm of the H- γ Al₂O₃ and H- β Co-Crystalline Zeolite. Zeolite sample as drop wise with mixing for 2hr. At 25°C. The mixture was left at room temperature for 24hr., it was stirred intermediately during this time. The mixture was then slowly evaporated to dryness over a period of 8 hr. By heating on a heat mantle. The resulting catalyst was dried in air at 110°C for additional 24hr. Then the dried catalyst was calcined at 400°C for 3hr. And reduced with hydrogen at 350°C for 2hr. (Satoshi, 2003) (Goodarz *et al.*, 2008) (Dhanapala *et al.*, 2008).

Analysis and Characterization: In order to know the Chemical composition of Zeolite, prepared samples were analyzed by X-ray Fluorescence (XRF) at geological research authority of Sudan and crystal structure the X-ray diffraction (XRD) pattern was recorded on Phillips X" Pert Pro PW 3719 X-ray diffractometer with Cu K α 1 and Cu K α 2 radiation source (λ =1.54056 Å and 1.54439 Å) respectively. Slits width 1/8 and 1/4 have been applied. Tension=40 kV, Current=30 MA. The range of angles scanned was (0 to 80) on 2 θ . At petroleum laboratories, research & studies of Sudan. To determine the morphology of zeolite, the scanning electron micrograph (SEM) was obtained by ZEISS microscope and Gold/Palladium coated sample used Inca X-Stream and mix software from Oxford Instruments Company. Energy Dispersive X-Ray (EDAX) it connected with the (SEM) to measure the composition of the zeolite Analysis at University of Khartoum Electron Microscope Unit, the surface area is calculated from the Brunauer-Emmett-Teller (BET) equation based on the physical adsorption of an inert gas at constant temperature, usually nitrogen at the temperature of liquid nitrogen. The principle of measurement consists in determining the point when a monomolecular layer of gas covers the surface of the catalyst.

RESULTS

γ -Al₂O₃ and β Co-Crystalline Zeolite chosen with different compositions as shown in table 1, and X-ray Diffraction (XRD) shown in figure 2, and figure 3, Scanning Electronic Microscopy (SEM) shown in Figure (4) and Figure (5) and Energy Dispersive X-ray (EDAX) shown in Figure 6 and Figure 7, the

Brunauer-Emmett-Teller (BET) shown in Table (3).

Table 1: Composition of γ -Al₂O₃ and β Co-Crystalline Zeolite

Composition	Al ₂ O ₃	SiO ₂	TiO ₂	Fe ₂ O ₃	SnO ₂	PtO ₂	P ₂ O ₅	K ₂ O	La ₂ O ₃	L.O.I
γ -Al ₂ O ₃	93.041	0.528	0.426	0.406	0.617	0.532	4.450
β Co-Crystalline Zeolite	51.179	41.210	0.316	0.595	2.142	0.389	1.31	2.859

Table 2: Surface Area and Pore Volume of γ -Al₂O₃ and β Co-Crystalline Zeolite

Catalysts	Surface Area m ² /mg	Pore Volume cm ³ /gm
Pt/H- γ -Al ₂ O ₃	288.86	0.3199
Pt/ H- β Co-Crystalline Zeolite	427.98	0.6603

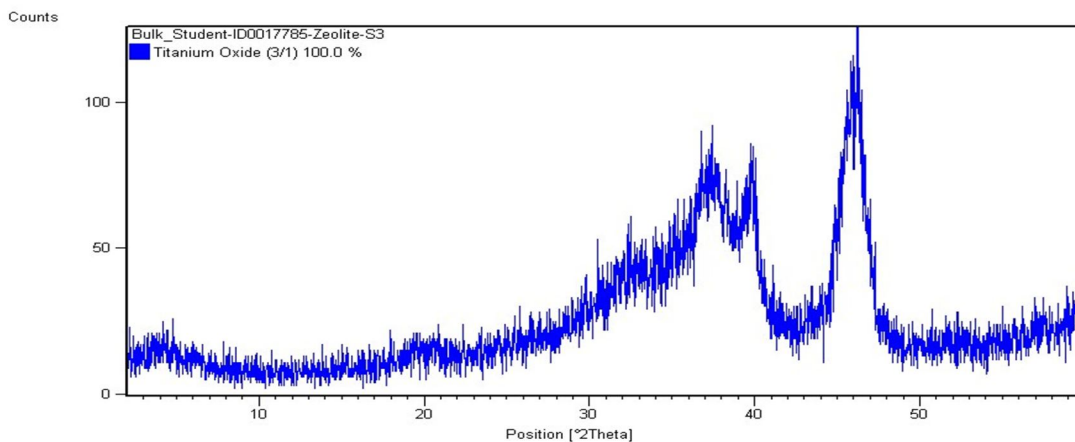


Figure 2: XRD patterns of γ -Al₂O₃

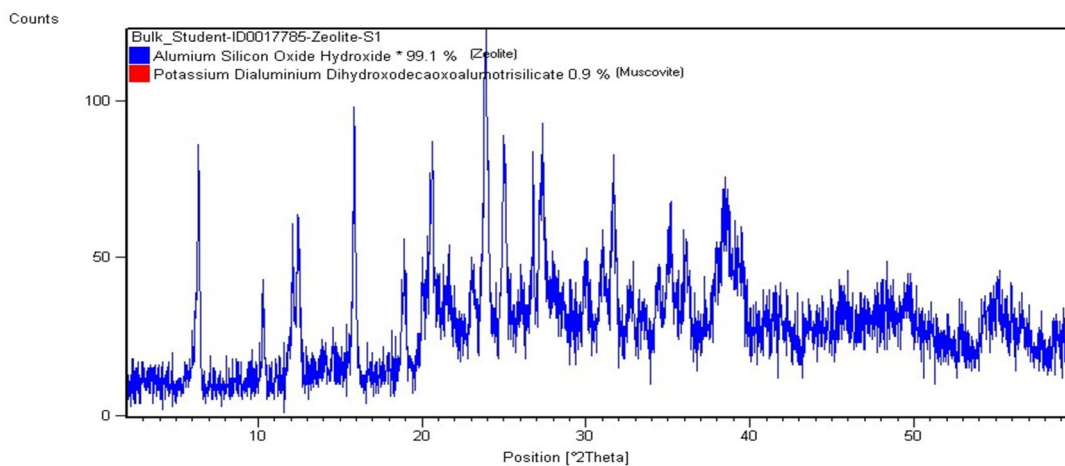


Figure 3: XRD patterns of β Co-Crystalline Zeolite



Figure 4: SEM monograph of Pt/H- γ -Al₂O₃

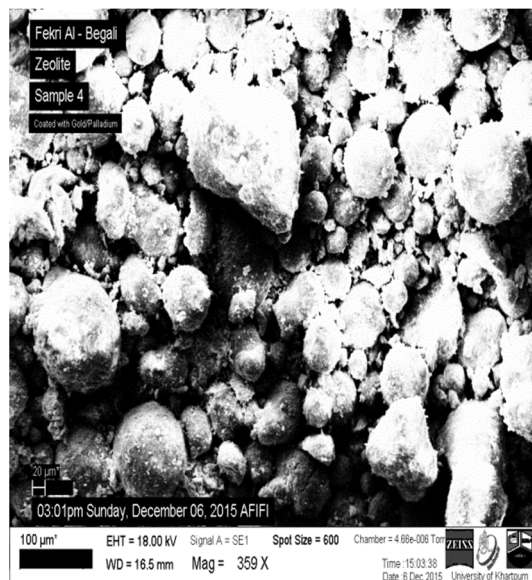


Figure 5: SEM monograph of Pt/H- β Co-Crystalline Zeolite

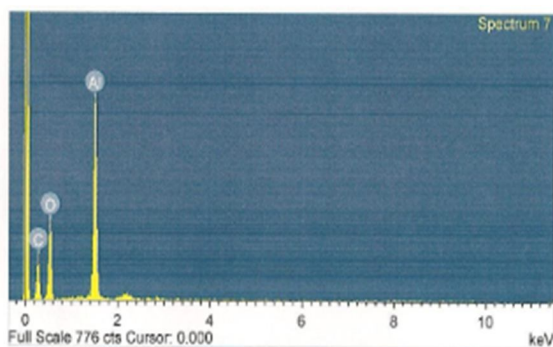


Figure 6: (EDAX) Pt/H- γ -Al₂O₃

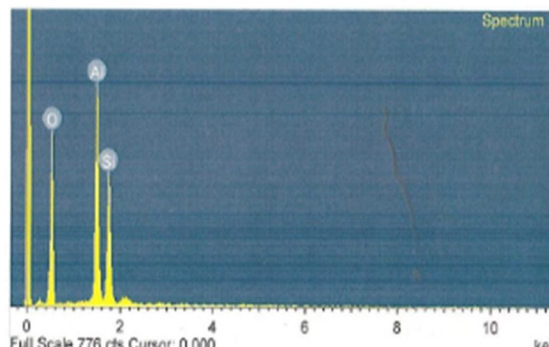


Figure 7: (EDAX) Pt/H- β Co-Crystalline Zeolite

DISCUSSION

Characterization might be studied or controlled in terms of supported properties, metal dispersion and location and surface morphology (Manoj *et al.*, 2012). Composition of Catalysts before preparation in table 1 (XRD) patterns was determined in figure 2 and figure 3 which shown pattern different phases and average crystalline sizes also shown

γ -Al₂O₃ amorphous Aluminum Oxide with Titanium Oxide crystallite and β Co-Crystalline Zeolite Aluminum Oxide and Silicon Oxide 99.1% crystallite with Potassium Dialuminium DiHydroxide 0.9% Muscovite, after preparation Characterization of Catalysts, (SEM) were used to determine the morphology and average crystallite size of the catalysts. Figure 4 and Figure 5 shown

the (SEM) monograph of pt./H- γ -Al₂O₃ and pt./H- β Co-Crystalline Zeolite respectively. Platinum particles were seen homogeneously distributed, where the white spots represent a platinum particle and black zone represent the supported, with the average diameter of the pt/H- γ -Al₂O₃ catalyst is 7 μ m, while for pt/H- β Co-Crystalline Zeolite catalyst is 8.5 μ m. These results are in accordance with that the faujasite crystallite size range (5-10) μ m. SEM is used to ensure good impregnation of active components. Also, from Figure 5 and figure 6 it is clear that the Al main components of pt/H- γ -Al₂O₃ and the Al and Si are the main components of pt/H- β Co-Crystalline Zeolite catalyst structure where the Si/Al molar ratio is equal to approximately 1.98. This molar ratio is calculated depending on the where the composition measurement inside the zeolite structure and pores at different positions in the structure. (BET) were the results of Surface Area giving the highest values equivalent 288.86m²/mg and 427.98m²/mg for Pt/H- γ -Al₂O₃ and Pt/ H- β Co-Crystalline Zeolite respectively and Pore Volume equivalent 0.3199cm³/gm 0.6603cm³/gm respectively. Where noted, the catalysts giving the highest values of the surface area and pore volume.

CONCLUSION

The Characterization of pt/H- γ -Al₂O₃ and pt/H- β Co-Crystalline Zeolite were studied after preparation by ion exchange and loaded with 0.5 wt. % Pt by impregnation with an aqueous solution of Hexachloroplatinic acid (H₂PtCl₆.6H₂O) methods where get on the modified catalyst Pt/ H- γ -Al₂O₃ and Pt/ H- β Co-Crystalline Zeolite. They are often also referred to as molecular sieves Zeolite with acid sites. Strong acidity

with a fine distribution must be the most important property observed in the zeolite catalyst. Because the thermal and mechanical stabilities are enough to use in the industrial process, and the zeolite synthesis requires to decrease much cost and time, so many kinds of zeolite species are not available industrially, these are modified zeolite adequately can be used in a variety of applications such as petrochemical cracking, Hydroisomerization and other applications.

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