Alumina doped with Fe synthesized by sol gel: Characterization and trichloroethylene catalytic combustion

Javier Rivera De la Rosa[1,2]* Araceli Hernandez[1], Carlos J. Lucio Ortiz[1] and José Antonio De los Reyes Heredia[3]

[1]*Facultad de Ciencias Químicas, Universidad Autónoma de Nuevo León, Av. Universidad s/n, Cd. Universitaria, San Nicolás de los Garza, N.L. C.P. 64450, (México).*

[2]*Centro de Innovación, Investigación y Desarrollo en Ingeniería y Tecnología (CIIDIT), Km 10 de la nueva carretera al Aeropuerto Internacional de Monterrey, PIIT Monterrey, C.P. 66600, Apodaca, Nuevo León, (México).*

[3]*Departamento de Ingeniería Química, Universidad Autónoma Metropolitana-Iztapalapa, P. O. Box 55-534, México D. F. 09340, (Mexico*)

*javier.riverad@uanl.edu.mx

Introduction

The sol–gel method has attracted considerable attention for the preparation of metallic catalysts, since the constituents are mixed in an atomic scale and this generates a uniform distribution of active metals upon the support [1]. The crystalline phase obtained by the sol–gel method depends on the starting metallic precursor. The samples obtained this way were thoroughly characterized with the purpose of evaluating and comparing them as useful catalysts for the combustion of trichloroethylene. The importance of studying the synthesis of new catalysts for destruction of trichloroethylene molecule is that it belongs to the group of chlorinated volatile organic compounds (VOC) that can be see a simulation of a pollution of air by a industrial gas emission.

In a past work; zirconia synthesized by sol-gel doping with La and Fe showed that crystallization temperature and structural characteristics are affected by metallic doping with La and Fe agents. The catalyst resisted the high concentration of trichloroethylene flow (1% wt in air) and developed a better performance compared with pure zirconia catalyst in the corresponding catalytic test due to the presence of tetragonal zirconia phase [2].

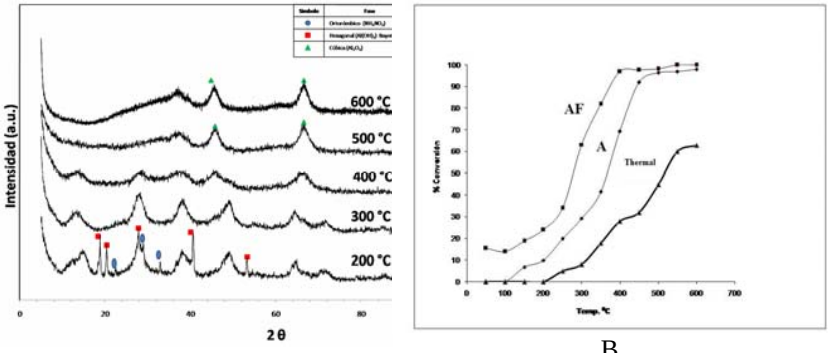
In this presenting research present the results of the synthesis of alumina pure and doped with Fe at 5 % wt , that were used as catalyst powders and tested both in a fix bed of 1 g tubular continues reactor. The catalysts were characterized during the synthesis by different techniques in order to establish a correlation between the morphology and physical-chemical properties with their performance in the surface combustion reaction of a chlorinated VOC in air.

Materials and Methods

For the synthesis, $(Al(NO_3)_3{\cdot}9H_2O)$ was dissolved in water and NH_4OH 8 M solution was added under magnetic stirring until reaching pH ~ 9.5. $Fe(NO_3)_3$ solution was added dropwise to the colloidal alumina; the doping iron represented a 0.5 wt. % of the Al_2O_3 mass. The solution was maintained under refluxing conditions at 25 °C until a gel was formed. Xerogels were then obtained by heating the gel samples at 67°C followed by calcination in an electrical furnace at 600 °C for 8 h. The powders obtained were pure alumina (A catalyst labeled) and alumina doped with Fe 0.5% wt. (AF labeled). Both powders were characterized by FT-IR, TGA, DTA, XRD and SEM techniques; the microstructure of pores was determined from N_2 adsorption isotherms The catalytic activity was tested using a steady state tubular micro-reactor inserted in a tubular furnace equipped with a temperature controller, employing mixtures of trichloroethylene in air (3200 ppm by volume) and the spatial time was of 500 hr^{-1}.

Results and Discussion

The annealing temperature of gels was determined according to TGA results; the volatile compounds were totally evaporated at 400 ° C but the temperature of calcination was increased until 600C in order to protect the catalytic tests. The cubic phase (γ-alumina) was favored by the doping agent. Figure 1(a) presents the x-ray diffraction patterns of doped sample at different temperature.



a B

Figure 1. (a)XRD patterns of iron doped alumina (AF catalyst) at different (b) Combustion of trichloroethylene at 3200 ppm in air for A and AF catalysts, thermal combustion is showed at the same conditions.

The X-ray diffraction pattern of the AF sample shows that at 200 ºC is still presents the precursors of sol gel synthesis. At 400 ºC is starting to define the cubic phase of alumina (γ-alumina), and at 600 ºC it is well formed. The specific surface areas calculated by BET method were 254 m^2/g for pure alumina (A catalyst) and 239 m^2/g for AF catalyst. In the combustion test (Figure 1 b), it is observed that at 600 ºC the 100% of conversion for both catalysts is reached. The selected temperature of calcination was the optimums to develop this test. It is distinguished in the thermal combustion (the reactor charged with inert silica) the conversion of trichloroethylene begins upper 200 ºC nevertheless for A catalyst the conversion at the same temperature is 9 % and for AF catalyst is 21 % at the same temperature. It can be state that both synthesized catalyst, pure and doped alumina, exhibit catalytic activity, depending on the surface reaction taking place at a range from 50 at 200 ºC.

Significance

It is presented the novel catalyst for combustion of volatile organic compounds in particularly chlorinated specie.

References

1. T. Lopez, M. Alvarez, R. G´omez, D.H. Aguilar, P. Quintana J. Sol–Gel Sci. Technol. 33 (2005) 93–97.
2. De la Rosa, J. R., Hernández A., Rojas F., Ledezma J. J. Colloids and Surfaces A: Physicochem. Eng. Aspects 315 (2008) 147–155