Comparative Analysis For Pseudoboehmite Obtained By Sol Gel Using Green Chemistry Expert System software

G. Camargo*. J. Serna**, J,C Moreno***, A. F Suarez****

*Universidad Libre Mechanical Eng. Dept Gabriel.camargov@unilibrebog.edu.co
**Universidad de los Andes. Chemical Eng Dept tulita_sesa@hotmail.com
***Universidad de los Andes. Chemistry Dept. jumoreno@uniandes.edu.co
****Universidad Libre. Environmental Eng. Dept. andresf.suareze@unilibrebog.edu.co

ABSTRACT

. The research was aimed to analyze whether the aging time and type of catalyst used in obtaining isopropóxide have any effect on the textural characteristics of the solid (surface area, pore size and volume) obtained in the end. The initial alkoxide was processed by the technique proposed by BE Yoldas. The reactants were analizated by infrared and the product were characterizated by TGA, XRD, surface area, pore volumen and pore size. The results of area, pore size and volume were analyzed with the software Desing Expert, to determine whether those factors had influence in these three variables. Finally, environmental analysis was performed comparing the two methods of synthesis of aluminum alkoxide in order to know the amount of waste produced by each of the catalysts and thus to establish which of the two is more environmentally friendly . For this purpose the simulation package Green Chemistry Expert System was used.

Keywords: sol gel method, pseudoboehmite, TGA, Green Chemistry

1. INTRODUCTION

The chemical industries are innovating to fix theirnprocess to new challengues. The ceramic industries provide elaboration methods such as sol gel method which is possible to make membranes, aerogel, etcand other items with unique properties such as pseudoboehmite (1,2,3). In this research, pseudoboehmite was synthesized using two different catalysts and comparing the enviromental impacts using Green Chemnistry Expert System software.

Monohidróxide aluminum, better known as pseudoboehmite (AlOOH) was obtained by the sol-gel method by the reaction between aluminum and isopropyl alcohol to produce aluminm isopropoxide, using two different catalysts; mercuric iodide and Iodine sublimed

2. MATERIALS AND PROCEDURES

For pseudoboehmite synthesis, the first step was reaction

$$Al + 3(IPA)OH \rightarrow Al(OR)_3 + \frac{3}{2}H_2$$

Where IPA is Isopropylic alcohol (JT Baker), the aluminiun (Merck) and mercuric iodide and Iodine sublimed were used as catalysts. Al $(OR)_3$ is aluminum isopropoxide. The reaction temperature was 353 K.

The next steps in the pseudoboehmite synthesis was the hydrolysis process by the following reactions:

$$M - O - R + H_2O \rightarrow M - OH + R - OH \quad \textbf{Ec. 1}$$

$$M - OH + OH - M \rightarrow M - OM + H_2O \quad \textbf{Ec. 2}$$

$$M - O - R + HO - M \rightarrow M - O - M + R - OH \quad \textbf{Ec. 3}$$

The peptization is the next process after hydrolysis. In this step HNO_3 was used as peptizating agent. At the end of this step an aluminum sol is obtained. The sol is dried (363 K) to evaporated water and nitric acid and form an aluminum gel. This gel is aging.

This procedure is based in a experimental design with type of catalyst was a category factor and aging time as numeric factor,

3. RESULTS

The results obtained in this project are shown in the next tables and figures:

Aging Time	Catalyst	surface area (m^2)
4h	I_2sublimed	371.4
1h	I_2sublimed	336.5
7h	I_2sublimed	380.0
7h	I_2sublimed	373.3
1h	HgI_2	363.9
7h	HgI_2	397.1
4h	HgI_2	367.2
4h	I_2sublimed	376.1
4h	HgI_2	381.8
1h	HgI_2	343.0

Table No. 1 Surface area obtained for two catalists and aging time

Aging Time	Catalyst	Pore size(Å)
4h	I_2sublimed	180.8
1h	I_2sublimed	193.3
7h	I_2sublimed	171.2
7h	I_2sublimed	171.1
1h	HgI_2	174.5
7h	HgI_2	173.8
4h	HgI_2	167.6
4h	I_2sublimed	167.3
4h	HgI_2	176.0
1h	HgI_2	174.7

Table No.2 pore size obtained for two catalists and aging time

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	2214,32	3	738,11	6,36	0.0271	significant
A-t aging time	2166,22	1	2166,22	18,67	0.0050	
B-Catalist	282,70	1	282,70	2,44	0.1696	
AB	23,45	1	23,45	0,20	0.6688	
Residual	696,28	6	116,05			
Lack of Fit	337,81	2	168,90	1,88	0.2651	not significant
Pure Error	358,48	4	89,62			
Cor Total	2910,60	9				

Table No. 3 ANOVA analysis of the experimental design

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	8,90E-04	3	2,90E-04	4,97	0.0458	
A-t aging time	7,50E-04	1	7,50E-04	12,57	0.0121	
B-Catalyst	2,80E-04	1	2,80E-04	4,75	0.0722	
AB	3,70E-05	1	3,70E-05	0,63	0.4582	
Residual	3,60E-04	6	6,00E-05			
Lack of Fit	2,20E-04	2	1,10E-04	3,47	0.1335	not ignificant
Pure Error	1,30E-04	4	3,30E-05			
Cor Total	1,20E-03	9				

Table No. 4 ANOVA for pore size

3.1 ENVIROMENTAL ANALYSIS

The reaction realizated with iodine sublimed is more friendly with the enviroment because this catalist produce 50.22% of total residues while the other catalist produce 58.34 % of total residues. This result are shown in the figures 1 and 2.

The principal inconvenient with Iodure HgI_2 is the formation of elemental mercury at the end of the first reaction..

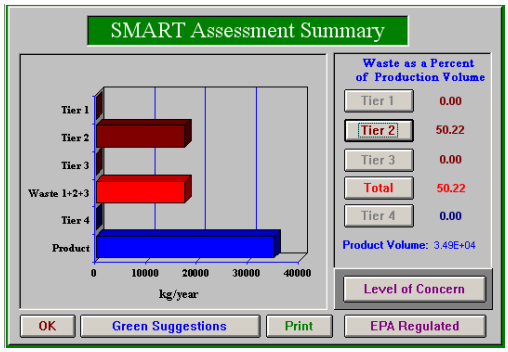

Figure 1/. General Result with I_2 Sublimed

Clean Technology 2011, www.ct-si.org, ISBN 978-1-4398-8189-7

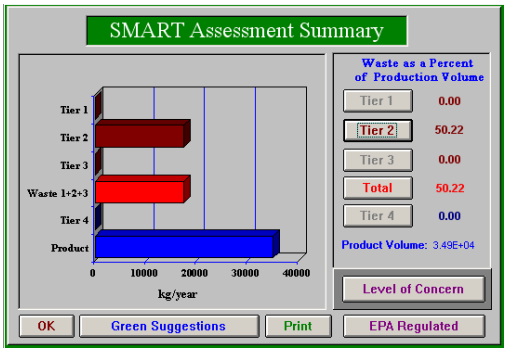

Figure 2. General Result with HgI_2

4. CONCLUSIONS

The type of catalysts does not have a significance effects on the textural properties of the pseudoboehmite obtained. The aging time has an influence on the .superficial area of the pseudoboehmite and the I_2 is friendlier with the environment according to GCES results.

The results shown that the surface area of the pseudoboehmite finally obtained is only going to be affected by the aging time of alumina gel, the longer time, the bigger the area tends to be. By contrast, the type of catalyst used in the reaction of alkoxide has no impact on this variable. The pore volume is going to be affected by aging time, but not by the type of catalyst. As the time increases the pore volume tends to increase. The catalyst is used as I_2 sublimated is more environmentally friendly than HgI_2. The experimentals results and the software simulation shown that amount of isopropoxide produced is higher with I_2 sublimated than HgI_2.

REFERENCES

[1]. B.E. Yoldas. **Alumina sols preparation from alkoxides.** Ceramics Bulletin (1975), Volume 54, Pages 289-290.

[2]. B.E. Yoldas. **Hydrolysis of aluminum alkoxides and bayerita conversion.** Journal of applied chemistry and biotechnology (1973), Volume 23 Pages 803-809.

[3]. S. Marciano, N. Mugnier, P. Clerin, B. Cristol and P. Moulin. **Nanofiltration of Bayer process solutions** Science Direct, Journal of membrane science (2006) Volume 281 Pages 260-267/