

Investigation of Salt and precipitating agent effect on the specific surface area and compressive strength of alumina catalyst support

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Nowadays, catalyst supports are extensively used to decrease the costs and increase the contact surface area in chemical reactions. Specific surface area, compressive strength, pore volume and pore size are some of the most important characteristics of a catalyst support. In this work, Sol-gel and peptization methods were applied to produce alumina catalyst support. Also the roles of aluminum salts and precipitating agents on the specific surface area and compressive strength of alumina catalyst support were investigated. In addition, various additives and common methods in the increasing surface area, compressive strength and adjusting the porosity and pore size are used in this study. The results show that using caustic soda as precipitating agent and aluminum chloride salt yields catalyst supports with the best compressive strength. Also, using aluminum nitrate and ammonia as precipitating agent produced alumina catalyst support with the highest specific surface area.

Keywords: Alumina, Catalyst Support, Sol Gel, Specific Surface Area, Compressive Strength.

INTRODUCTION

Having a high available contact surface and economic advantages causes applying catalyst supports, to be enhanced in recent years. Alumina is among the catalyst support, which have been very frequently used in recent decades for many reactions, due to its extended range of specific surface area, compressive strength, pore volume and pore size, and its various phases¹. Alumina catalyst support has many applications in the steam reforming^{2,3}, desulfurization^{4,5}, oxidation of toluene⁶, methane and propane dehydration^{7,8}, hydrogen production^{9,10}, removal of the bacteria and odor gases¹¹, hydrogenolysis of glycerol¹², and also in automobile exhaust catalytic converter and other petrochemical processes^{21,22}. Extended applications of alumina as a catalyst or a catalyst support cause many scientists to do a lot of researches on producing and developing them. Many studies in these fields have also been done by using peptization^{12,13} and Sol-gel methods¹⁴⁻¹⁸. In addition, many researches have been conducted to investigate the effect of the considerable factors in the process, indicating time, temperature and pH of the reaction have significant influence on properties of catalyst supports produced by Sol-gel method¹⁹. Some studies show that calcination conditions have important effects on the characteristics of the alumina catalyst support²⁰. Recent studies also revealed that the initial concentration of aluminum has a major impact on specific surface area, pore volume and pore size of the alumina catalyst support²³⁻²⁶. The pH of the solution is the another important parameter in producing alumina catalyst support²⁷. The results show an increase in pH from 5 to 7, decreases the specific surface area and pore volume. It also increases the pore size of the catalyst support²⁷. Another study indicates that an increase in pH from 4 to 5 reduces all of these parameters²⁸. The reaction temperature has also a remarkable influence on the catalyst support properties²³. In addition, using various surfactants is an effective factor on the proper-

ties of alumina catalyst support^{27,29-31}. Some researchers have exposed that time and temperature of the aging step is one of the most important parameters in this process³²⁻³⁵. These results indicate that the specific surface area and the pore size are decreased and increased with escalation of aging time, respectively³⁶. The calcination step condition is one of the most important issues in the catalyst support production process. Recent studies showed the type of the alumina that is formed in the process mainly depends on the calcination temperature³⁷⁻⁴⁰. The investigations revealed that calcination temperature had a major influence on the properties of the alumina catalyst support^{23,29,30,41}. Increasing the calcination temperature reduces the specific surface area, the pore volume and the pore size^{33,42-44}. The results showed that the biggest surface area was produced in temperature range of 400–450°C⁴⁵. The time of calcination is another significant parameter in the process. There is a reduction in the specific surface area when the calcination time is raised^{32,35,46}. The speed of the temperature change is also an effective parameter on the characteristics of the alumina⁴⁶.

This study has been conducted to produce alumina catalyst support, using Sol-gel and peptization methods. In the Sol-gel method, aluminum nitrate, aluminum sulphate and aluminum chlorid were used as the aluminum sources. Ammonia, caustic soda, sodium carbonate and sodium aluminate liquor have been used as the alkaline precipitating agents. The roles of the various aluminum sources and precipitators on the specific surface area and compressive strength were investigated, too. In the peptization method, aluminum hydroxide was applied and the effects of HNO₃ on the specific surface area and compressive strength of alumina catalyst support were investigated.

Material and compounds analysis

An electric furnace (Carbolite-RW1400, England), a dryer (Ecocell, England) and a heater equipped with

temperature control and magnetic stirrer were used to perform the experiments. The quantitative analyses of the compounds in the aluminum hydroxide and alumina were performed using XRF (Siemens-SRS3000, German) and AAS (Unicam, England) devices. The required equipment for measuring the compressive strength has been made. The BET surface area was measured using Strohlein-Aramat II.

The required aluminum nitrate, aluminum sulphate, aluminum chlorid, ammonia, caustic soda, sodium carbonate, HNO₃ have been provided from Merck Company products. Aluminum hydroxide (Table 1) produced by Jajarm Alumina Company in Iran was applied for the tests. In addition, the required sodium aluminate solution was prepared and produced using caustic soda from Merck Company.

Table 1. Chemical and physical analysis of the aluminum hydroxide

Chemical	SiO ₂ [%]	Fe ₂ O ₃ [%]	Na ₂ O [%]	CaO [%]	L.O.I [%]	Moist [%]
	0.007	0.006	0.31	0.008	34.77	9.19
Physical	Attrition index		<20 μm	<44 μm	<150 μm	Median
	%		%	%	%	μm
	3.0		1.1	10.3	17.8	101.4

DESCRIPTION OF THE EXPERIMENTS

Sol-gel method

Sol-gel method was used with the aim of investigating the roles of the various aluminum sources and precipitating agents on the specific surface area and compressive strength (Table 2). Each aluminum salt was separately mixed with different precipitating agents and stirred for 0.5 hr. In the experiments pH was kept constant at

Table 2. Aluminum sources and precipitating agents

Experiment No.	Aluminum Salt	Precipitating agent
1	Aluminum Nitrate	Ammonia
2		Caustic soda
3		Sodium carbonate
4		Sodium Aluminate
5	Aluminum Sulphate	Ammonia
6		Caustic soda
7		Sodium Carbonate
8		Sodium Aluminate
9	Aluminum Chloride	Ammonia
10		Caustic soda
11		Sodium Carbonate
12		Sodium Aluminate

9 and then the produced gel was aged for 20 hours at 25°C. In the next step the produced white hydro gels were filtered, washed and then in the next step, the granulated aluminum hydroxide, having the diameter

of 4–7 mm, was made using liquid paraffin. Granulated product was washed and dried at a temperature of 110°C for 2 hours. In the calcinations step, the dried balls were calcined at the temperature of 550°C for 4 hours. In this step, the temperature change rate was set to 5°C/min (Table 3 and Fig. 1).

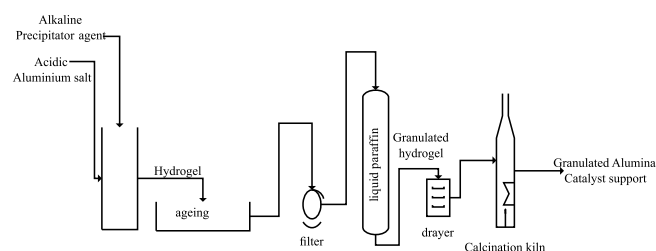


Figure 1. Diagram of the process of alumina catalyst support production, using the Sol-gel method

Specific surface area and compressive strength of produced alumina catalysts were measured using Strohlein-Aramat II and BET nitrogen adsorption. For measuring compressive strength, the grain of catalyst was placed on a surface and a mobile piston, operated by a stepping motor, crushes the grain. And an electronic device measures the peak value when the grain breaks.

Peptization method

This study also used peptization of aluminum hydroxide in the producing alumina catalyst support. In the first step, 100 g of hydrated alumina was heated at 700°C for about 0.5 minute and then mixed with water to produce slurry with solid content of 500 g/l. Then produced slurry was treated using nitric acid. The weight ratio of nitric acid to the alumina hydrate in the experiment was 2.5:1000. This mixture was stirred at 70°C for 6 hours. Then the hydrated product was dried at 110°C. In the next step, the produced hydrated alumina was mixed with distilled water to prepare the slurry with the concentration of 250 g/l (alumina hydrate). This suspension was mixed with nitric acid. Molar ratio between nitric acid and aluminum hydroxide was 0.05. The produced slurry has been stirred at 90°C for 4 hours. Then treated suspension was filtered and washed to form the granulated alumina hydroxide. Drying and calcination conditions were the same as those for the Sol-gel method conditions.

Table 3. Operation conditions of the producing alumina catalyst support steps, using the Sol-gel method

Reaction step			aging step		drying step		Calcinations step		
Temp [°C]	pH	Time [h]	Temp [°C]	Time [h]	Temp [°C]	Time [h]	Temp [°C]	Temp rate [°C/min]	Time [h]
25	9	0.5	25	20	110	2	550	5	4

RESULTS AND DISCUSSION

Sol-gel method

As mentioned above, the production conditions have direct influences on characteristics of the alumina. Therefore, in the experiments, these conditions were kept constant as presented in Table 3 and various acidic aluminum salts and alkaline precipitators have been used to produce alumina catalyst support. Specific surface area, compressive strength and the Catalyst support diameter are given in Table 4.

Table 4. Characteristics of the Sol-gel method products

Exp. No	Specific surface area [m ² /g]	Compressive strength [kg/cm ³]	Catalyst support diameter [mm]
1	300	0.5	4.0
2	209	1.1	7.6
3	96	0.4	6.5
4	164	9.0	6.5
5	51	0.2	4.0
6	245	14.9	4.6
7	61	2.0	5.0
8	176	2.0	8.0
9	208	0.3	4.8
10	154	20.1	3.5
11	73	2.1	6.0
12	236	2.0	8.3

Effect of various alkaline precipitators on alumina catalyst support characteristics

Investigating the precipitator type on the specific surface area and compressive strength of the alumina catalyst support has been performed in this study. These results are given in the Figures 2–7.

Effect of various precipitators on specific surface area in the presence of aluminum nitrate

Aluminum nitrate salt and various precipitators have been used to produce alumina catalyst support. Specific surface area of these products were measured and presented in Figure 2. Figure 2 shows that ammonia produces the highest specific surface area (300 m²/g) whereas sodium carbonate causes the lowest specific surface area (96 m²/g). The results also indicate using sodium hydroxide and sodium aluminate solutions lead the specific surface area of 209 and 164 m²/g, respectively.

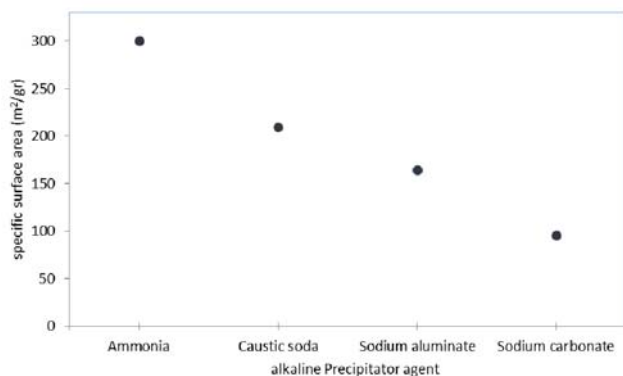


Figure 2. The effects of various alkaline precipitators on the specific surface area in alumina catalyst support produced by aluminum nitrate

Effect of various precipitators on specific surface area in the presence of aluminum sulfate

Figure 3 shows the specific surface area of alumina catalyst supports, which were made from aluminum sulfate. In this case, the carrier produced from sodium hydroxide solution with specific surface area of 245 m²/g, has the highest value whereas the carrier produced from ammonia causes the catalyst supports has the lowest specific surface area of 51 m²/g.

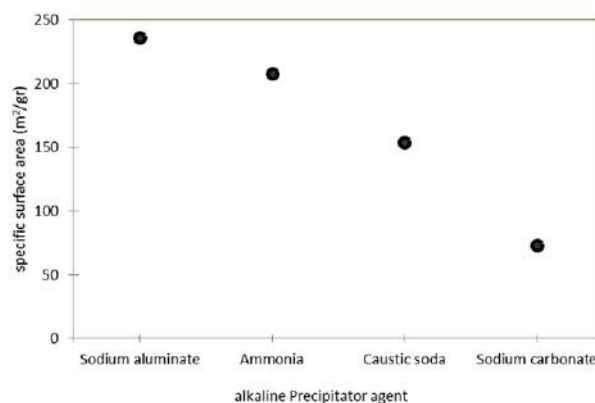


Figure 3. The effect of various alkaline precipitators on the specific surface area of the alumina catalyst support produced from aluminum sulfate

Effect of various precipitators on the specific surface area in the presence of aluminum chloride

Figure 4 shows the alumina catalyst support produced using aluminum chloride salt and 236 m²/g sodium aluminate as the precipitating agent leads to the largest value of specific surface area compared to other precipitators. These results also indicate that sodium carbonate leads the catalyst carrier with the lowest BET.

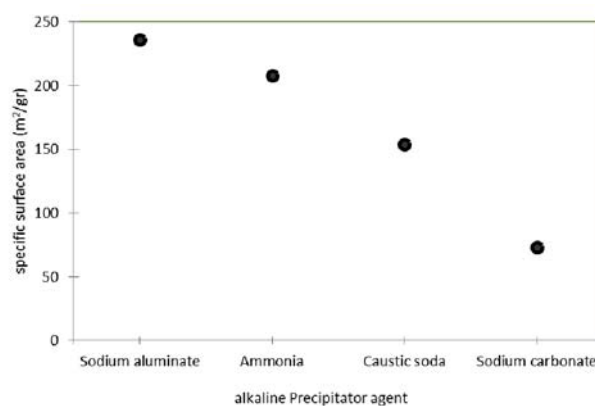


Figure 4. The effect of various alkaline precipitators on specific surface area of alumina catalyst support produced by aluminum chloride

Effect of various precipitators on compressive strength in the presence of aluminum nitrate

Figure 5 shows the results of the investigation of the various alkaline precipitators and acidic aluminum sources effects on compressive strength of alumina catalyst support in the presence of aluminum nitrate. The results indicate the alumina produced using sodium aluminate has the greatest compressive strength compared to others. The Figure also shows sodium carbonate has the lowest compressive strength among the precipitators.

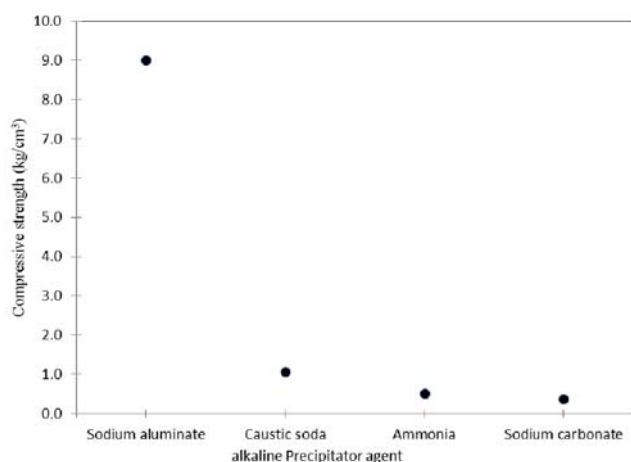


Figure 5. The effect of various alkaline precipitators on compressive strength of alumina catalyst support produced by aluminum nitrate

Effect of various precipitators on compressive strength in the presence of aluminum sulfate

Figure 6 shows the compressive strength of the alumina catalyst supports that were produced from aluminum sulfate salt and the various precipitators. The results indicate that sodium hydroxide can produce alumina catalyst carrier with the highest compressive strength compared to the other precipitators. The Figure also shows that the catalyst carrier prepared using ammonia has the lowest compressive strength among various precipitators.

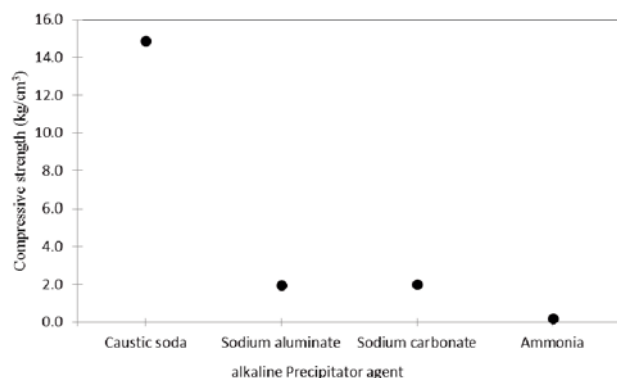


Figure 6. The effect of various alkaline precipitators on compressive strength of alumina catalyst support produced by aluminum sulfate

Effect of various precipitators on compressive strength in the presence of aluminum chloride

Figure 7 shows using aluminum chloride salt and sodium hydroxide as the precipitator has produced alumina catalyst support with compressive strength of 20.1 kg/cm³. That means its compressive strength is much higher than other carriers. This Figure also exposes that alumina catalyst support produced applying ammonia has the lowest amount of compressive strength (0.3 kg/cm³).

Peptization method

In this study, peptization method has also been used to prepare alumina catalyst support. In the treating process, nitric acid is also been applied. Table 5 shows the alumina catalyst carrier specific surface area, compressive strength and the catalyst support diameter. The results show that peptization method causes the alumina to have specific

Table 5. Characteristics of the alumina catalyst support produced by peptization method

Experiment No	specific surface area [m ² /g]	Compressive strength [kg/cm ³]	Catalyst support diameter [mm]
13	200	0.3	0.4

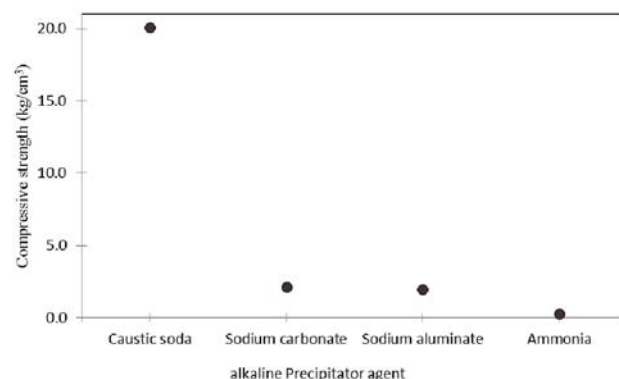


Figure 7. The effect of various alkaline precipitators on compressive strength of alumina catalyst support produced by aluminum chloride

surface area of 200 m²/g. It is very important to mention that in this study no additive has been used to increase BET and compressive strength. In this condition as shown in Table 5, the compressive strength is 0.3 kg/cm³.

Comparison of the effects of various aluminum salts and precipitators on specific surface area and compressive strength

Comparison of the effect of various aluminum salts on the specific surface area and compressive strength using average amount of these data in the presence of different precipitators are shown in Figure 8. The results show that the alumina catalyst carriers has been produced using aluminum nitrate and aluminum sulfate with specific surface area of 192.25 m²/g and 133.25 m²/g, respectively. The study of the impacts of using various aluminum salts on compressive strength indicates that the catalyst support prepared from aluminum chloride and aluminum nitrate leads to the maximum and minimum amounts of compressive strength.

The average amount of specific surface area and compressive strength of aluminum catalysts supports produced from various alkaline precipitators are presented in Figure 9. The results show that alumina catalyst supports produced using sodium hydroxide and sodium carbonate

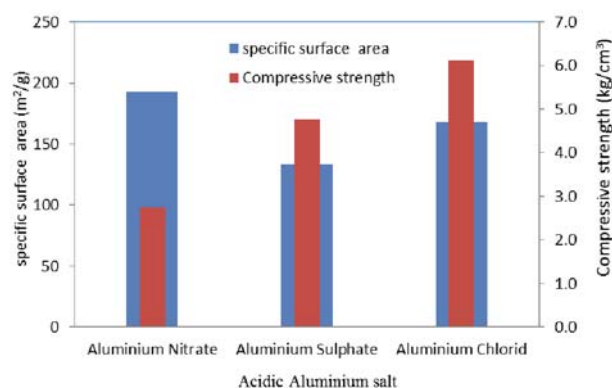


Figure 8. The effect of various aluminum salts on compressive strength and specific surface area of alumina catalyst support

with specific surface area of 202.7 m²/g and 76.7 m²/g have the highest and the lowest values, respectively. Also, sodium aluminate has been used to prepare the catalyst support with a high surface area. This study indicates that various precipitators have different influences on the compressive strength. Figure 9 reveals caustic soda and sodium carbonate applied to make alumina catalyst supports with the highest and the lowest compressive strength. These results also show that sodium aluminate causes the product to have high compressive strength.

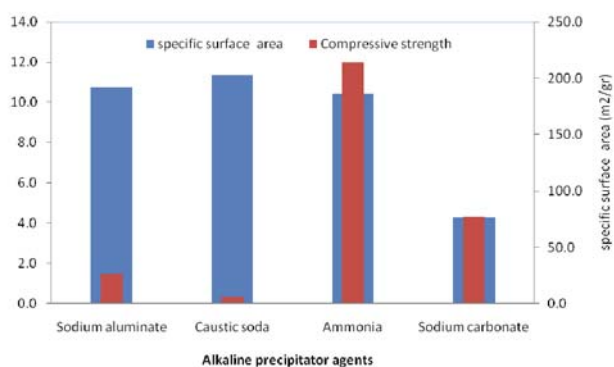


Figure 9. The effect of various alkaline precipitators on compressive strength and specific surface area of alumina catalyst support

All comparison results of the effects of various aluminum salts and precipitator agents on the specific surface area and compressive strength is summarized and presented in Table 6.

Table 6. Effects of various aluminum salts and precipitator agents on the specific surface area and compressive strength

Aluminum salts	Specific surface area	Compressive strength
	[m ² /gr]	[kg/cm ³]
Aluminium Nitrate	192.25	2.7
Aluminium Sulphate	133.25	4.8
Aluminium Chlorid	167.75	6.1
Alkaline precipitators	Specific surface area	Compressive strength
	[m ² /gr]	[kg/cm ³]
Sodium carbonate	76.7	1.5
Ammonia	186.3	0.3
Caustic soda	202.7	12.0
Sodium aluminate	192	4.3

CONCLUSION

This research has achieved appropriate pairs of salt and precipitator for producing alumina catalyst supports with the large of specific surface area and compressive strength. The results show that based on Sol-gel method, aluminum nitrate salt along with the ammonia as a precipitating agent produced alumina catalyst support with 300 m²/g of specific surface area. The catalyst carrier with the highest compressive strength of 20 kg/cm³ has been prepared from aluminum chloride and sodium hydroxide. The results also indicate that alumina catalyst supports prepared from aluminum nitrate and aluminum chloride have the largest specific surface area and compressive strength, respectively. Among various precipitators in the presence of different aluminum sources exposes, sodium hydroxide and sodium aluminate have produced the supports with the highest

values BET of 202.7 and 192 m²/g, respectively. These results also indicate that sodium hydroxide causes the carrier to have the largest compressive strength compared to the other precipitating agents. Furthermore, in the study, peptization method has been used to produce alumina catalyst support with specific surface area of 200 m²/g and compressive strength of 0.3 kg/cm³.

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