Sakarya University Journal of Science

ISSN : 2147-835X
Publisher : Sakarya University

Vol. 28, No. 1, 304-313, 2024
DOI: https://doi.org/10.16984/saufenbilder.1323558

Research Article

The Effect of Peptizing Agent Concentration on Processing and Properties of Alumina Based Catalyst Support

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ARTICLE INFO

ABSTRACT

Keywords:
Catalyst Support
Gamma Alumina
Peptizing
Extrusion

Gamma alumina (γ-Al2O3) has drawn attention as a support material for heterogeneous catalysts extensively used in the oil and gas industry due to its superior properties. Boehmite (AlOOH) is an industrially accepted and well-known precursor to produce gamma alumina-based support. The process additions such as nitric acid (HNO3) are commonly used in shaping process of alumina to control textural, mechanical and structural properties of final catalyst support. In this work, gamma alumina precursor boehmite was peptized by nitric acid at molar ratio of HNO3/AlOOH ranging from 0 to 0.054. With addition of nitric acid, extrusion paste showed higher plasticity up to 0.017 mole of HNO3/AlOOH. The addition of more than this point led to an inhomogenity of extrusion paste caused by hard and brittle structure caused by the formation of aluminum nitrate salts. Also, higher mechanical strength was observed for samples peptized with lower nitric acid concentration because of effective deagglomeration by peptization. The pore size of catalyst support pellets increased up to an acid/ boehmite molar ratio of 0.017 by peptizing of boehmite. However, beyond this point pore size decreased due to dissolution of boehmite.

Article History:
Received: 06.07.2023
Accepted: 23.12.2023
Online Available: 22.04.2024

1. Introduction

In modern industry, more than 90% of processes are catalytic [1]. Many important processes require heterogeneous catalysts in which the reactant phase and catalyst phases are different. Heterogeneous catalysts are among the main solutions for sustainable industrial applications [2]. Catalysts basically consist of two parts: a support material and micro or nano-sized active metals positioned on this support material [3]. The primary function of the catalyst support material is to create a surface to site to the active metal parts and to increase the area of the active parts [4]. Among all the catalyst support materials, γ-Al_2O_3 is in the class of transition alumina and is widely used in the oil and gas industry thanks to its superior properties such as cheapness and abundance, chemical and mechanical inertness, and controllable acid-base characteristics [5]. Boehmite (AlOOH) is an important precursor for the preparation of γ-Al_2O_3 catalysts because the heat treatment of boehmite produces a series of transition alumina from γ-Al_2O_3 and η-Al_2O_3 to δ-Al_2O_3, and θ-Al_2O_3, which exhibit high surface areas (200–500 m^2/g) [6]. Thus, boehmite is the most frequently used precursor to γ-Al_2O_3 support. Understanding and optimizing the properties of the support material is crucial for current and future studies and one of the ways to improve the final performance of the catalyst is to improve the properties of the support material. One of the most important factors for optimizing the catalyst material is the shape of the catalyst. The shape of the catalyst has a great influence on the process

Cite as: S. Turan, I. S. Ökten, C. Açıksarı, S. Çelebi, U. Savacı, E. Ayas (2024). The Effect of Peptizing Agent Concentration on Processing and Properties of Alumina Based Catalyst Support, *Sakarya University Journal of Science,* 28(2), 304-313. https://doi.org/10.16984/saufenbilder.1323558

(flow regime, pressure drop, etc.) and performance [7]. Extrusion is the most widely used shaping technique for forming fixed bed catalytic support materials [8].

To achieve extrusion successfully, process additions influence the extrusion process and final product properties [9]. One of these process additives is the peptizing agent in alumina systems and is necessary to prevent agglomeration of powders by dispersion of boehmite [10]. Boehmite particles used as starting material of γ-Al_2O_3 support are usually supplied in agglomerated form. The agglomerated particles provide easier and cleaner handling, a more manageable production environment, and improved storage qualities. However, it is necessary to break agglomerates to shape and tune the properties of catalyst. Alumina based catalysts are commonly formed by peptization of boehmite agglomerates by dilute monoprotic acids (e.g., HNO_3) [11].

The acid as peptizing agent helps break agglomerates into smaller particles as much as possible. This leads to favorable extrusion by improving the plastic behavior of the paste [12, 13]. Although several patents and publications deal with extrusion of alumina with nitric acid as a peptizing agent, this detailed study based on the effect of wide range of HNO_3 concentration on the final properties of γ-Al_2O_3 that is used as a catalyst support in oil& gas refinery [14, 15]. γ-Al_2O_3 catalysts are also shaped to optimize mechanical requirements and peptizing agents help to increase to a certain point [16, 17]. Furthermore, design of tailored textural properties (surface area, pore size and pore distribution) requires a good knowledge of the effect of the peptization process [18].

The detailed analyses were related to the deagglomeration phenomena of boehmite particles by using nitric acid solution. The aim of this work was to investigate the effect of a wide range of peptizing agent concentration on extrudability and properties of γ-Al_2O_3 catalyst support.

2. Methods

2.1. Materials

Boehmite powder (Sasol GmbH, Germany) having BET surface area of 280 m^2/g and the average particle size of 40 μm, nitric acid (HNO_3, ≥ 65%, Sigma-Aldrich) as peptizing agent and deionized water were used in the present investigation.

2.2. Catalyst support shaping

Catalyst support pellet for the extrusion was carried out by the following procedure. First, boehmite powder was mixed with optimum amount of nitric acid at various concentrations and only deionized water until having a homogeneous mixture prior to extrusion for 16 min at 40 rpm by means of a mixer (Caleva Multi Lab, UK) with Z-shaped mixing arm. The nitric acid with different concentrations was added slowly to the boehmite particles to obtain a plastic dough for extrusion. The molar concentrations of nitric acid and its corresponding HNO_3/ AlOOH by mole and HNO_3/ total composition by wt. were calculated and given in Table 1. The solid content for each batch was adjusted to the level resulting in the highest torque.

The solid content by wt. percent for each batch was given in Figure 1. The optimum solid content decreased with increasing HNO_3 concentration due to effect of gelation caused by the deagglomeration of boehmite particles. Then, paste was transferred to a single screw extruder (Caleva Multi Lab, UK) for shaping through a 2 mm diameter die at an extrusion speed of 30 rpm. The green extrudates were dried at 80 ^oC for overnight in an oven and cut into the length of 2-4 mm sized pellets. Schematic flow chart for pellet preparation was summarized in Figure 2. After, they fed into the the box furnace where they were calcined at 600 ^oC for 5 h with a heating rate of 3 ^oC/ min to obtain gamma alumina.

Table 1. HNO_3/ AlOOH and HNO_3/ total composition by wt. of various HNO_3 molar concentrations

Molar concentration of HNO_3 solution (M)	HNO_3/ AlOOH (mole/mole)	HNO_3/ total composition (% wt.)
0.00	0.000	0.00
0.10	0.004	0.44
0.20	0.009	0,90
0.25	0.011	1.14
0.37	0.017	1.69
0.50	0.025	2.47
0.75	0.037	3.63
1.00	0.054	5.15

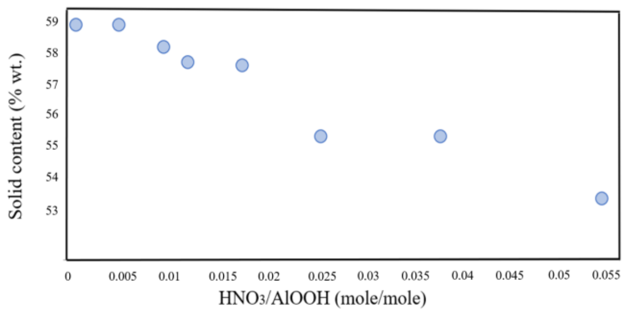

Figure 1. The solid content (wt. %) versus HNO_3/AlOOH (mole/mole) of each batch

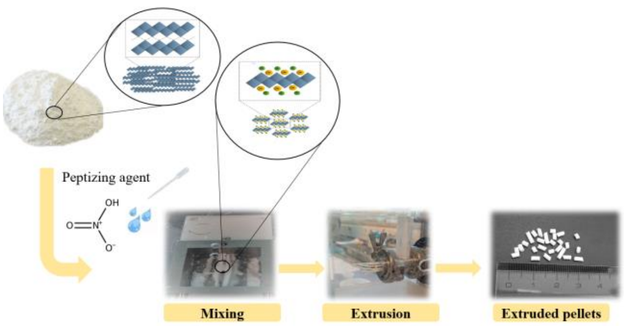

Figure 2. Schematic flow chart for pellet preparation by extrusion of the dough consisting of boehmite and nitric acid

2.3. Characterization

Rheological characterization of the paste was determined by using a mixer torque rheometer-MTR (Caleva, MTR-3, UK). The optimum solid content of mixture and mix time of paste for extrusion were analyzed for each batch.

The morphology of as-received boehmite particles was carried out via scanning electron microscopy (SEM- Zeiss Supra 50VP, Germany).

The thermal behavior of the green extrudates was conducted on a thermogravimetric analyzer (Perkin-Elmer, TGA 4000, USA) at a heating rate of 5 °C/min under air atmosphere. The crystal phases of boehmite powder and the extrudates were determined at 2θ= 10°-80° with a scanning speed of 1°/min by using an X-Ray diffractometer (XRD- Bruker, D8 Advance, Germany) with Cu Kα X-ray source.

The strength of extrudates was determined by crushing test method (VINCI Technologies, France). Single pellet strength was determined by uniaxial compression between two parallel rigid platens. The diameter of every pellet was measured with a caliper before the pellet was placed on the bottom plate of a press. The crushing strength is calculated by dividing the maximum recorded load by diameter. At least 25 pellets are tested and then all tested samples are averaged and calculated standard deviation.

Brunauer-Emmett-Teller- BET (Micromeritics, ASAP 2020, USA) gas sorption measurement was performed to investigate the specific surface area and porous structure of the extrudates. After 6 hours of degassing at 300 °C, multi-point BET analysis was performed based on the N_2 gas adsorption technique in a liquid N_2 at 77.3 K. The pore size distribution and pore diameters were obtained by a Barett–Joyner–Halenda- BJH model from the desorption branches of the isotherms.

3. Results and Discussion

3.1. Identification of the starting powder

The boehmite powder was used as precursor; its properties were summarized in Table 2. It is a member of the alumina hydrate family and was supplied as a spray-dried particle. The final crystalline phase and physical properties of calcined gamma alumina depend on the initial crystalline properties of the starting precursor as well as the calcination temperature [19]. Boehmite transforms into γ-Al_2O_3 transition alumina under a temperature range of 450–650 °C with a departure of structural water [20]. The sequence of phase transitions was given in Figure 3 [21].

Table 2. Properties of Boehmite-AlOOH powder

Properties	Boehmite powder
Al_2O_3 (wt. %)	74
Na_2O (wt. %)	0.002
Particle size d_{10} (μm)	10
Particle size d_{50} (μm)	40
Particle size d_{90} (μm)	85
Packed bulk density (g/l)	800-1100
Surface area (BET) (m^2/g)	280
Pore volume (BET) (cm^3/g)	0.43
Pore diameter (BET) (nm)	5
Crystallite size (120)* (nm)	5
Loss on ignition (LOI) (wt. %)	26

*The size is calculated by Scherrer equation for (120) plane

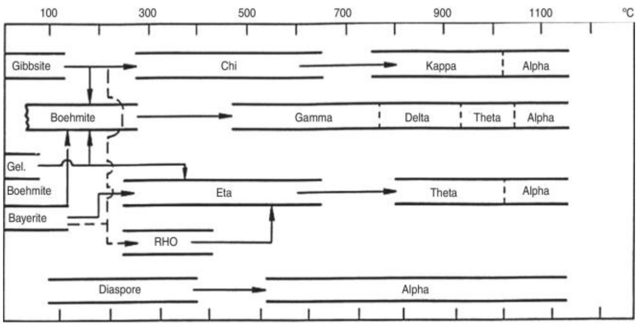

Figure 3. Transformation sequences of precursors to α-$A1_2O_3$

The variation of zeta potential of boehmite vs pH in aqueous media was demonstrated in Figure 4 [22]. As it can be seen, the zeta potential and repulsive force between the boehmite particles are high at pH<8 due to surface charge effect. Furthermore, stable region in acidic zone is wider than basic. This is the reason for both nitric acid and water are able to peptize the boehmite particles.

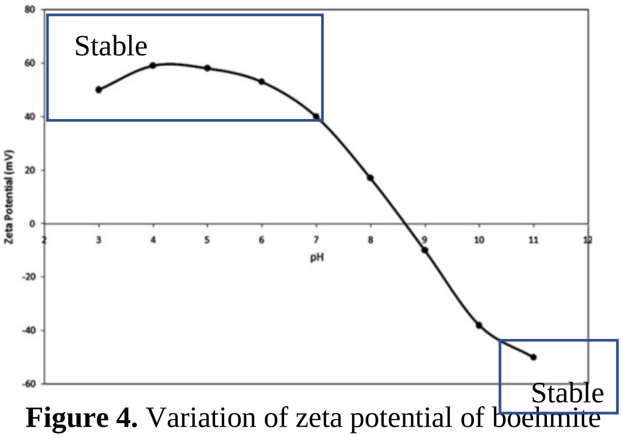

Figure 4. Variation of zeta potential of boehmite versus pH in aqueous media

As already mentioned, supplied boehmite is a spray-dried and the presence of large particles and agglomerates were detected by using SEM images in Figure 5.

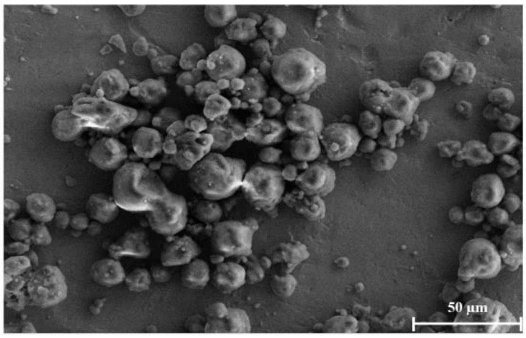

Figure 5. SEM images of as received boehmite

3.2. Rheological characterization

Mixer torque rheometer (MTR) has been widely used to determine the optimum amount of liquid in formulations intended for extrusion and spheronisation [23]. MTR was used to characterize the rheological properties of batches containing varying amounts of HNO_3 and water. The result of mean line torque (Nm) vs binder ratio (ml/g) of the boehmite powder peptized with only water was given in Figure 6. According to Figure 6., 40 rpm mixing rate was chosen for extrusion for all mixtures due to its highest torque value. Also, optimum binder ratio 0.533 ml/g was used while solid content of mixtures was studied.

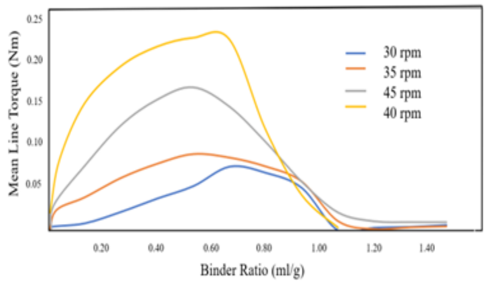

Figure 6. The mean line torque (Nm) versus binder ratio (ml/g) of the boehmite powder peptized with only water

The effect of peptizing agent concentrations on torque of wet mixtures was given in Figure 7. It results in the maximum torque value increased with increased peptizing agent molar ratio. However, the mixtures were not favorable after a certain point for extrusion. In Figure 7, the mixture below was favorable for extrusion and the mixture above was not. Although the torque was high, extrudability of the mixture was quite low at high concentrations of acid.

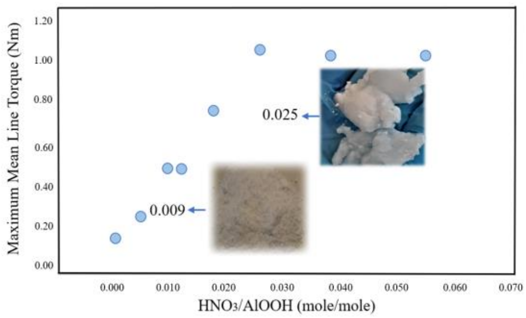

Figure 7. The maximum mean line torque (Nm) versus HNO_3/AlOOH (mol/mol) values of each batch

The HNO_3/AlOOH ratio is critical for extrusion of alumina. If the extrusion paste does not have sufficient consistency, the shape of extrudates differs. The extrudates stuck with each other at low ratios of the HNO_3/AlOOH and they were very glassy and brittle at high ratios of the HNO_3/AlOOH. The pictures of stuck, ideal, glassy extrudates were given in Figure 8.

Figure 8. Images of the extrudates peptized with (a) 0 HNO_3/AlOOH, (b) 0.017 HNO_3/AlOOH, (c) 0.054 HNO_3/AlOOH molar ratios

(b)

3.3. Thermal analysis

TG graph of shaped green sample peptized with various HNO_3/AlOOH molar ratio was seen in Figure 9. The all green extrudates showed similar thermal behavior. The large mass change corresponds to the conversion step from boehmite to γ-alumina, and it is also attributed to the removal of chemically adsorbed water from the structure. In the last step, although no thermal event occurs for mixture. There is a continuous mass loss starting at about 600 °C. Here, residual hydroxyls are removed from the structure and the mass loss is 1.85 wt. % [7]. Although the amount of the acid concentration increases, it is not observed a significant change in the temperature of boehmite to gamma alumina transition However, it is stated in the literature that high amount of nitric acid leads to the formation of basic aluminum salts between primary particles of boehmite [24, 25]. Consequently, the weight loss in TGA analysis corresponds to the removal of as formed aluminum salts at the samples that were prepared by adding a higher amount of HNO_3.

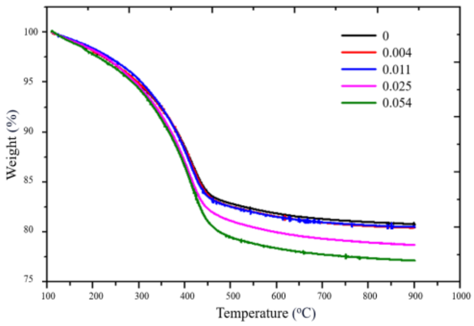

Figure 9. TG graph of the green sample peptized with various HNO_3/AlOOH molar ratios

3.4. XRD analysis

XRD graph of boehmite powder not peptized at room temperature and after peptization with HNO3/AlOOH ratio and calcined at 600 °C was given in Figure 10. It was seen that the obtained diffraction pattern for boehmite powder at room temperature belongs to regular orthorhombic boehmite (JCPDS file of 21–1307). After calcination at 600 °C for 5 h, all green extrudates transformed to gamma alumina phase. Gamma alumina crystals have been conventionally described as a defect spinel (cubic spinel structure with presence of tetragonal distortion along one of the axes of the spinel cell) (JCPDS file of 10-0425) [6]. According to the literature, it is known that a significant change in crystallite shape and size cannot be made with the addition of acid, but only in their spatial arrangement [15]. This is in agreement with XRD results showing that there were no significant changes in characteristic peaks of gamma alumina which produce at different nitric acid concentrations.

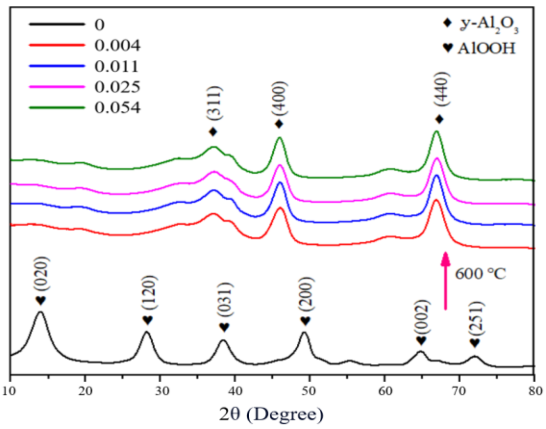

Figure 10. XRD patterns of boehmite powder not peptized at room temperature and after peptization with HNO_3/ AlOOH ratio and calcined at 600 °C

3.5. SEM images

Secondary scanning electron microscopy images (showed in Figure 11) were taken from the cross-section of extrudates supports produced by0.00, 0.017 and 0.054 HNO_3/AlOOH mole ratio and calcined at 600 °C in air atmosphere. A large agglomerate of Alumina particles in micron size was seen in Figure 11a due to the absense of peptizing agent. In Figure 11b, it was seen that increase in acid content leads to a breaking of boehmite agglomerates by chemical attack. A denser packing microstructure is clearly observed on a sample prepared with 0.054 HNO_3/AlOOH mole ratio. This result is also in good agreement with the pore size distribution results calculated from N_2 adsorption/desorption isotherm in Table 3. It can be explained by the peptization process involving a partial dissolution of boehmite particles starting from particle surface [24, 25].

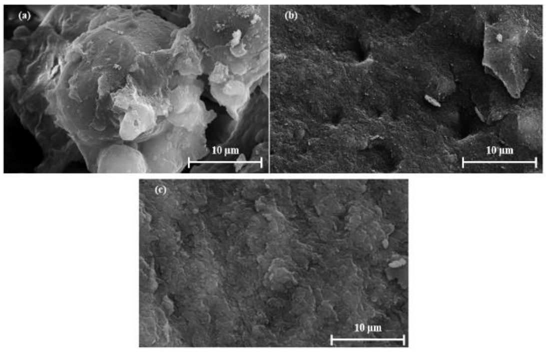

Figure 11. SEM images of calcined samples peptized with different HNO_3/AlOOH (mole/mole) ratios: (a) 0; (b) 0.017; (c) 0.054

3.6. Mechanical properties

The graph of crushing strength versus HNO_3/AlOOH (mole/mole) of the extrudates was given in Figure 12. The extrudates with highest strength were around 26 N/mm at the HNO_3/AlOOH ratio of 0.009. It was followed by 25 N/mm at the HNO_3/AlOOH ratio of 0.012 and 22 N/mm at 0.017. The peptization of boehmite by nitric acid up to 0.017 HNO_3/AlOOH mole ratio, breaks down the coarse soft agglomerates of primary particles and increase plasticity of pastes, which enhances mechanical strength of the final catalyst support.

Hovever, it is known that an excess amount of nitric acid leads to dissolution of primary pseudoboehmite particles more and resulting in low paste consistency during extrusion process [24]. Therefore, the nitric acid molar ratios greater than 0.017 were not found to be suitable due to having insufficient compression strengh. Samples that were produced by HNO_3/AlOOH molar ratios between 0.009 and 0.017 were a good stability and stiffness under mechanical loads for a high-performance catalyst support especially when compared to the samples that were peptized with the distilled water (10.78 N/mm) and highest amount of HNO_3 (6.54 N/mm).

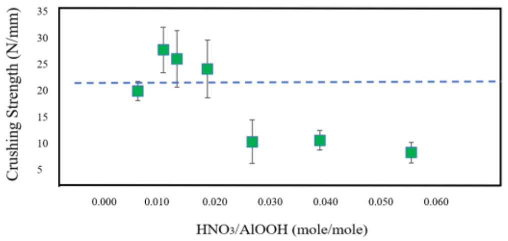

Figure 12. The crushing strength vs $HNO_3/AlOOH$ (mole/mole) of the extrudates

3.7. Textural properties

The adsorption-desorption isotherms of extrudates peptized with different $HNO_3/AlOOH$ molar ratios were given in Figure 13. The type IV hysteresis on the adsorption and desorption behavior of all samples peptized with different molar ratios confirmed the mesoporous structure.

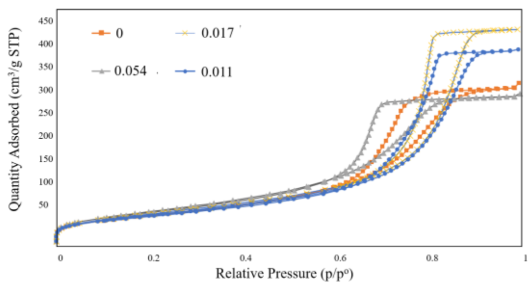

Figure 13. Nitrogen adsorption-desorption isotherm (Langmuir Type IV) of calcined samples peptized with various $HNO_3/AlOOH$ (mole/mole) ratios

The pore diameter (Å) and pore volume (cm3/g) of extrudates versus $HNO_3/AlOOH$ (mole/mole) was presented in Figure 14. According to the figure, with increasing molar ratio $HNO_3/AlOOH$ up to 0.017, pore diameter and pore volume increased due to peptizing effect. Then, they decreased dramatically when acid/alumina ratio of extrudates exceeded 0.017. For calcined samples prepared with relatively higher nitric acid content ($HNO_3/AlOOH$= 0.025, 0.037 and 0.054), the small pore diameter and pore volume are consistent with their corresponding close-packed morphology (Figure 11c) and partial dissolution of the initial aluminum hydroxide would lead to smaller porosity.

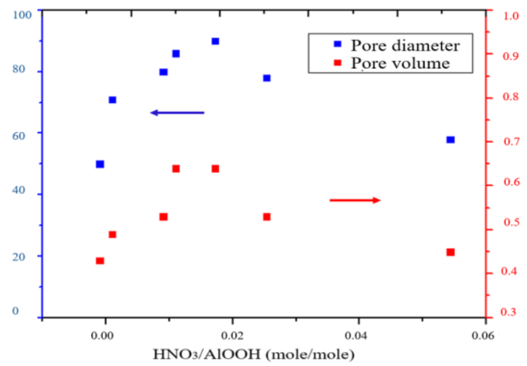

Figure 14. Pore diameter (Å) and pore volume (cm^3/g) of extrudates versus $HNO_3/AlOOH$ (mole/mole)

Pore size distribution of calcined samples peptized with different $HNO_3/AlOOH$ (mole/mole) ratios were given in Figure 15. Even if the pore diameters change with nitric acid molarity, the pore size distribution of all prepared samples showed a uniform pore size in the range of 5–9 nm.

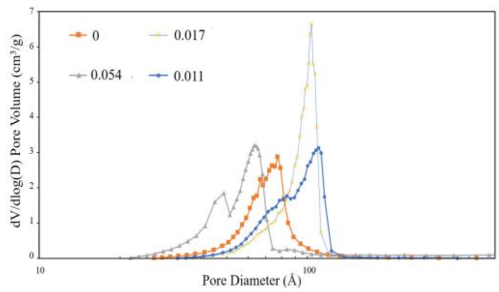

Figure 15. Pore size distributionof calcined samples peptized with various $HNO_3/AlOOH$ (mole/mole) ratios

The textural properties of the extrudates at various $HNO_3/AlOOH$ molar ratio were given in Table 3. BET surface area of as received boehmite exhibited the highest value. It was expected to reduce BET surface area value with the calcination process.

Table 3. Textural properties of the selected extrudates

HNO_3/ AlOOH (mole/mole)	BET surface Area, m^2/g	Average Pore Diameter, Å	Total Pore Volume, cm^3/g
Boehmite	280	50	0.43
0	217	71	0.49
0.009	216	80	0.53
0.011	215	86	0.58
0.017	220	90	0.64
0.025	220	78	0.53
0.054	240	58	0.45

4. Conclusion

This study provided fundamental understanding of the effect of wide range of HNO_3 concentration where HNO_3 is used as a peptizing agent for boehmite deagglomeration, extrusion processing and properties of γ-Al_2O_3 catalyst support.

Optimum nitric acid/ boehmite molar ratio range was determined as 0-0.017. The paste became glassy and heterogenous at higher molar ratios due to over-gelation. The solid content by weight decreased with increasing HNO_3 concentration for forming in extrusion. The crushing strength values were greater than 20 N/mm when nitric acid/boehmite molar ratio is in between 0.009-0.017. Pore size and pore volume of catalyst increased until 0.017 nitric acid/boehmite molar ratio and decreased after the point of 0.017.

Article Information Form

Acknowledgments
The authors would like to thank TUPRAS (Türkiye Petrol Rafinerileri A.Ş.) with a project number P1.20AP.03 for laboratory infrastructure.

Funding
The authors would like to thank TÜBİTAK (119C161- 2244 industrial Ph.D. program) for financial support.

Authors' Contribution
The authors contributed equally to the study.

The Declaration of Conflict of Interest/ Common Interest
No conflict of interest or common interest has been declared by the authors.

The Declaration of Ethics Committee Approval
This study does not require ethics committee permission or any special permission.

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