The Effects of SiO$_2$/Al$_2$O$_3$ and H$_2$O/Al$_2$O$_3$ Molar Ratios on SAPO-34 Catalyst in the Methanol to Olefin Process

Ehsan Kianfar$^{1,2}$

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Abstract
Informed through synthesis and characterization of NH$_3$ TPD, BET techniques, the current study investigated the right contribution of silicon and water content on a 9 sample SAPO-34 catalyst with different molar ratios. The final prepared SAPO-34 catalytic performed reaction for the Methanol to olefin process was processed and reported at 410 °C a reactor of fixed-bed. It was shown that catalysts with different SiO$_2$ /Al$_2$O$_3$ and H$_2$O/Al$_2$O$_3$ ratios demonstrated high ethylene and propylene selectivity, due to their intensified crystallinity and size of crystals (1.3 to 1.7 μm). The highest degrees of ethylene and propylene selectivity for 3 samples were 50.25, 21.20, and 48.44, 19.16 respectively. Samples with ratios of different that possess high density and acute acidic sites to deactivate rapidly. The high density of acute acidic sites promoted the ultimate response to saturated hydrocarbons and aromatics based on olefins’ hydrogen transfer to and resulted in the coke generation on the top layer of the catalyst.

Keywords Methanol to olefin · BET · Ethylene · Propylene

1 Introduction
SAPO-34 is formed by silicon substitution of the chabazite-type aluminophosphate framework resulting in Bronsted acid sites formation [1–9]. Chabazite is a three-dimensional molecular sieve with ellipsoidal cages of 6.7 A × 10 A interconnected via 8-membered ring windows with pore openings of 3.8 A × 3.8 A [10–16]. The adjustment of acidity, as well as shape selectivity to promote product selectivity and/or control coking behavior, are two key properties that help researchers modify the reactor performance of such acidic crystalline material [17–25]. The acidity of SAPO-34 molecular sieves would vary by their silicon content; however, the relation between the number of acid sites and the silicon content is not linear because it is controlled by at least two Si incorporation mechanisms into the SAPO-34 framework [26–33]. The structure acidity and catalytic performance of SAPO-34 directly depend on the number and distribution of Si in the framework, which is related closely to the Si incorporation mechanism and coordination environment [34–40]. Acidity adjustment can be considered as a way of delaying or decreasing the rate of deactivation of SAPO-34 [41–45]. The acidity of SAPO-34 molecular sieves could be varied through isomorphs substitution of captions, action exchange, and adjustment of Si/Al ratio [46–50]. Acidity adjustment of SAPO-34 through the Si/Al ratio regulation may be achieved easier and cheaper since it does not need any additional chemical materials [51–59]. However, there are extremely few publications on the role of water content on the morphology and size of the SAPO-34 crystals [60–67]. Furthermore, the simultaneous effects of SiO$_2$/Al$_2$O$_3$ and H$_2$O/Al$_2$O$_3$ on the synthesis gel have not been studied [68–75]. In this regard, this paper aims to investigate thoroughly the effect of the changing of SiO$_2$/Al$_2$O$_3$ and H$_2$O/Al$_2$O$_3$ on the morphology and structure of the SAPO-34 catalyst.

|$^{1}$ Department of Chemical Engineering, Arak Branch, Islamic Azad University, Arak, Iran

|$^{2}$ Young Researchers and Elite Club, Gachsaran Branch, Islamic Azad University, Gachsaran, Iran
2 Experimental

2.1 Sample Preparation

SAPO-34 catalysts were synthesized by a hydrothermal method using different ratios of SiO$_2$/Al$_2$O$_3$ and H$_2$O/Al$_2$O$_3$ at the temperature 463 K. The molar composition of the reaction mixtures is given in Table 1. SAPO-34 was made by stirring and adding sequentially H$_3$PO$_4$, deionized water, and aluminum source (aluminum isopropoxide) in a PE beaker. Then, the source of silicon (silica gel) was added in small amounts and after that, the templates were used. Tetraethyl ammonium hydroxide (TEAOH, 20 wt% in water, Merck) and morpholine (Merck) were used as the organic templates. The mixture was then placed in a 100 mL Teflon-lined stainless steel autoclave. The solution was then transferred to a 100 mL teflon-lined stainless steel autoclave at 463 K for 24 h. The solid product was recovered and washed six times by centrifuging. The calcination of samples was done in air at 550 °C for 5 h to remove organic templates [76–82].

The molar composition of the reaction mixtures is given in Table 1.

2.2 Catalytic Test

Figure 1 is an overview of the laboratory unit. After the catalyst restoration, a mixture containing methanol and water (30% by weight methanol solution, 99.9%, Merck) is pumped into the furnace. The feed then enters the reactor from the bottom of the furnace. After the reaction, the outflow from the reactor enters the condenser and lowers the temperature to prevent secondary reactions. At this point, water and heavy hydrocarbons become liquid and accumulate in the condenser. The residual gas stream is transferred to the GC for analysis or to the outside environment. In addition to the feed stream of methanol and water entering the reactor, there is another stream that is attached to the nitrogen gas capsule. Because of the time gap between the synthesis and the reactor test, nitrogen gas was used to dehydrate and restore the catalysts. This step was performed before the reaction at 550 °C with a flow rate of approximately 40 ml/min for one hour [35, 83–86]. To adjust the current intensity before injection one Flow meter bubble was used. The furnace used in this laboratory unit, which is used to supply the heat required for the reactor and to perform the MTO reaction, is a pipe furnace manufactured by Azar Furnace Company. The furnace has a length of 45 cm and an inner diameter of 4.5 cm. GC unit used in a laboratory unit, Hewlett-Packard 5890 Flame Ionization Detector (FID), equipped with Agilent J&W GS-alumina column measuring 30 μm* 0.53* mm 30 m, manufactured by Hewlett-Packard and for the detection of hydrocarbon compounds. In this system, it is not possible to measure and detect hydrogen, carbon dioxide, and carbon monoxide, so the weight% of these three compounds in the gas sample are calculated by the elemental mass balance of the identified gases [35, 87–90].

2.3 XRD Characterization

Figure 2 shows the results of XRD analysis to determine the composition and crystallization of synthesized catalysts by various ratios of SiO$_2$/Al$_2$O$_3$ and H$_2$O/Al$_2$O$_3$. According to the XRD patterns, the position and the intensity of the diffraction peaks of the synthesized samples, S2 and S3, confirmed the structure type SAPO-34 (CHA). The patterns and peak positions match the patterns reported for this structure [1, 2]. When low silicon content is used, the crystalline phase of synthesized sample S1 is found to be significantly different as it is seen in Fig. 2. The major product is SAPO-34 with the characteristic reflection at 20 ≈ 9.66°, 13°, 20.52° with the presence of minor one, i.e. SAPO-5 with the characteristic reflection at 20 = 6.58°. Depending on the different ratios of SiO$_2$/Al$_2$O$_3$ and H$_2$O/Al$_2$O$_3$ used for the synthesis, the degree of crystallinity of the investigated samples

| Catalyst | (H$_2$O/Al$_2$O$_3$) | Synthesis gel composition (molar basis) | Time (h) |
|----------|----------------------|----------------------------------------|----------|
| S1 85    | 1Al$_2$O$_3$:1P$_2$O$_5$:0.1SiO$_2$:0.5TEAOH,1.5MOR,85H$_2$O | 24 0.1 |
| S2 25    | 1Al$_2$O$_3$:1P$_2$O$_5$:0.53SiO$_2$:0.5TEAOH,1.5MOR,60.25H$_2$O | 24 0.53 |
| S3 120   | 1Al$_2$O$_3$:1P$_2$O$_5$:0.35SiO$_2$:0.5TEAOH,1.5MOR,120H$_2$O | 24 0.35 |
| S4 75    | 1Al$_2$O$_3$:1P$_2$O$_5$:0.17SiO$_2$:0.5TEAOH,1.5MOR,109.75H$_2$O | 24 0.17 |
| S5 25    | 1Al$_2$O$_3$:1P$_2$O$_5$:0.17SiO$_2$:0.5TEAOH,1.5MOR,60.25H$_2$O | 24 0.17 |
| S6 85    | 1Al$_2$O$_3$:1P$_2$O$_5$:0.35SiO$_2$:0.5TEAOH,1.5MOR,85H$_2$O | 24 0.35 |
| S7 50    | 1Al$_2$O$_3$:1P$_2$O$_5$:0.35SiO$_2$:0.5TEAOH,1.5MOR,50H$_2$O | 24 0.35 |
| S8 75    | 1Al$_2$O$_3$:1P$_2$O$_5$:0.53SiO$_2$:0.5TEAOH,1.5MOR,109.75H$_2$O | 24 0.53 |
| S9 85    | 1Al$_2$O$_3$:1P$_2$O$_5$:0.6SiO$_2$:0.5TEAOH,1.5MOR,85H$_2$O | 24 0.6 |
varied. Sample S3, prepared at ratios of SiO$_2$/Al$_2$O$_3$ = 0.35, and H$_2$O/Al$_2$O$_3$ = 120 possessed the highest crystallinity. Sample S5, prepared ratios of SiO$_2$/Al$_2$O$_3$ = 0.17 and H$_2$O/Al$_2$O$_3$ = 60.25 has a peak in 2θ = 6.58°. by examination the references, it can be attributed to the SAPO-5 catalyst. The reason for this combination seems to be a low ratio SiO$_2$/Al$_2$O$_3$ in gel for Sample S5 synthesis. The crystallinity percentage of the catalysts concerning the peaks and the area under the graph of the XRD calculated according to the intensity of the peaks and their subsurface, and the results are summarized in Table 2. The data in Table 2 show that in samples with a molar ratio of SiO$_2$ / Al$_2$O$_3$ Constant with increasing the amount of water in the catalyst, we will reduce crystallization except he cases of SiO$_2$ / Al$_2$O$_3$ = 35, which increases the water ratio by first reducing the amount of crystallization and then increasing it. Regarding the effect of water, it can be said that in the lowest and highest amount of water in the samples, the amount of crystallization decreases with increasing Si content, while in H$_2$O / Al$_2$O$_3$ ratio On average, as the number of water increases, the crystallization of the samples first decreases and then increases. A large amount of water in the synthesis gel leads to the diffusion of more material, resulting in irregular crystals with low crystallization. The highest crystallization percentage is related to sample S5, while if we are looking for

| Sample | Crystal size(µm) |
|--------|------------------|
| S1     | 1.5–2.2          |
| S2     | 2.4–3.4          |
| S3     | 2.2–3.1          |
| S4     | 0.9–1.7          |
| S5     | 1–1.5            |
| S6     | 1.2–2.2          |
| S7     | 1–1.9            |
| S8     | 1.7–2.7          |
| S9     | 1.5–3.4          |
the highest crystallization percentage among pure SAPO-34 samples, this sample of catalyst sample S5 with molecular ratios $\text{SiO}_2 / \text{Al}_2 \text{O}_3 = 0.53$ and $\text{H}_2\text{O} / \text{Al}_2 \text{O}_3 = 60.25$, That is, it is an example that has high Si content and low water content [44, 91–95].

2.4 SEM Characterization

The SEM image of the synthesized samples is presented in Fig. 3. The average crystal size of sample S2, S3 is approximately 3 μm. It is shown that sample S2 has more regular crystals. Sample S3 has an incomplete structure maybe there is more water content in the primary gel leads to imperfect networks. Sample S1, synthesized with ratios of $\text{SiO}_2 / \text{Al}_2 \text{O}_3 = 0.1$ and $\text{H}_2\text{O} / \text{Al}_2 \text{O}_3 = 85$, presents a smaller size, irregular shape in morphology, and impurities. Also, comparing between samples displayed that the cubic SAPO34 crystals S2 and S3 were well distributed and got more regular size than ones in the sample S1. Thus, the SEM studies endorsed that the morphology of the final product is highly dependent on the ratios of $\text{SiO}_2 / \text{Al}_2 \text{O}_3$ and $\text{H}_2\text{O} / \text{Al}_2 \text{O}_3$. The SEM images of the samples show that the morphology of all samples is cube-shaped as expected for SAPO-34. Sample S5 in addition

Fig. 2 XRD pattern of synthesized SAPO-34 with different Various Ratios of $\text{SiO}_2 / \text{Al}_2 \text{O}_3$ and $\text{H}_2\text{O} / \text{Al}_2 \text{O}_3$
to cube-shaped particles, contains objects with hexagonal geometry that are related to SAPO-34. XRD analysis also confirms the figures SEM for this sample S5. From the analysis of the SEM images, particle size is given in Table 2. The smallest crystal sizes were observed for sample 4 and the largest size was observed for sample S2, which has the highest percentage of crystallization among pure sapphire samples. The high crystallinity percentage in the larger size sample could be due to the presence of the silicone islands after a certain S1 ratio, it is formed and affects the degree and length of the bond T–O–T, the structure, and size of the crystals. In all cases, as the SiO$_2$ / Al$_2$O$_3$ ratio increases in constant water, the size of the crystals increases. Too much water in the samples leads to less decentralization, and the particles in the gel contribute more to the growth of crystals [3–5].

2.5 Infrared Spectral Analysis (FTIR)

Infrared spectra of the samples in the framework vibration frequency region are shown in Fig. 4. According to the spectra, it is obvious that there are no characteristic peaks of
the amorphous phase in samples S1–S9. The characteristic absorption bands of the SAPO-34, i.e. absorption peaks at 642 (T–O bending in D6 rings), 571 (T–O bending of the phosphorous tetrahedron), 530 (T–O bending of silicon and aluminum tetrahedrons), 480 (T–O bending of silicon tetrahedrons) cm⁻¹ are in good agreement with the published data [3, 4, 96–100]. All samples display an adsorption band at 3620 cm⁻¹ which is assignable to OH stretching vibrations originating from Si (OH) Al bridging hydroxyl group [5]. The hydroxyl groups that are active sites are stronger in sample S1 because of their low ratio of SiO₂/Al₂O₃. It can be seen that there was no considerable increase in the concentration of the strong acidic hydroxyl groups in the samples of S2 - S9, corresponding to varying the ratios of SiO₂/Al₂O₃ and H₂O/Al₂O₃.

2.6 BET Analysis

The catalysts show mesopore, micropore, total surface area, and total pore volume as represented in Table 3. Water molecules have the structural directing role for the mesopore of the catalyst. Water molecules also fill the empty volume of the structure and mechanically prohibit the crash of the framework. Increasing silicon content in the composition when the amount of water is constant, leads to an increase in the surface area of the catalysts, due to the decrease in the average size of the crystals. As the SiO₂ / Al₂O₃ ratio is constant, the increase in water in the catalyst composition reduces the surface area, which was predictable due to the increase in crystal size [5–8, 101–104]. sample S9 has the maximum area (693.70), due to the maximum crystallinity percentage (97.76) among the samples S1-S8 respectively.

![Fig. 4 FTIR pattern of synthesized SAPO-34 with different Various Ratios of SiO₂/Al₂O₃ and H₂O/Al₂O₃](image)

2.7 EDX Analysis

In order to determine the composition of the fabricated catalysts, we review the results of the EDX analysis. For the tested samples, the Si entered into the structure was calculated from the following equation.

\[
\text{Si incorporation} = \frac{(\frac{\text{Si}}{\text{Si}+\text{Al}+\text{P}})_\text{Product}}{(\frac{\text{Si}}{\text{Si}+\text{Al}+\text{P}})_\text{initialgel}}
\]

According to the general formula SAPO-34, which is \((\text{Si}_{x} \text{Al}_{y} \text{P}_{z}) \text{O}_{2}\) the molar components of Al, P and Si were calculated in the structure and are given in Table 4. In all samples, Si is attached to the structure more than one, except in sample S3 which has the highest amount of water. Examination of the results of Table 4 in constant Si ratio shows that as water increases in catalytic samples, the Si attached to the structure decreases, which is consistent with the results of other studies. However, at constant water content, Si increases in the samples, first and decreases then and there is a slight increase in the amount of Si introduced into the structure. The highest Si in the structure is related to sample S7 with molar ratios of Al₂O₃ / SiO₂ = 35 and Al₂O₃ / H₂O = 50. The presence of high Si in the final composition predicts the possibility of silicon islands and increased acidity. The lowest value of this quantity is related to sample S3 with molar ratios of Al₂O₃ / SiO₂ = 35 and Al₂O₃ / H₂O = 120. Therefore, it can be said that the presence of too much water in the composition of the initial catalyst gel due to the reduction of supersaturating leads to more.

### Table 3 BET for prepared catalyst

| Sample  | Surface area, m²/g | Pore size, Å | Pore volume, cm³/g |
|---------|-------------------|-------------|-------------------|
| SAPO-34(S1) | 508               | 18.9        | 0.22              | 0.08 | 0.33 |
| SAPO-34(S2) | 555               | 19.0        | 0.18              | 0.10 | 0.29 |
| SAPO-34(S3) | 585               | 18.9        | 0.23              | 0.07 | 0.30 |
| SAPO-34(S6) | 627               | 19.8        | 0.20              | 0.03 | 0.23 |
| SAPO-34(S7) | 627               | 21.2        | 0.26              | 0.06 | 0.32 |
| SAPO-34(S9) | 694               | 18.7        | 0.27              | 0.07 | 0.32 |

### Table 4 Results of EDX analysis

| Sample  | Si_x  | Al_y  | P_z  | Si incorporation |
|---------|-------|-------|------|------------------|
| S1      | 0.0295 | 0.5367 | 0.4338 | 1.21              |
| S3      | 0.0646 | 0.5253 | 0.4101 | 0.8               |
| S6      | 0.0832 | 0.5310 | 0.3958 | 1.03              |
| S7      | 0.1288 | 0.2167 | 0.6544 | 1.6               |
| S9      | 0.1376 | 0.4998 | 0.3626 | 1.05              |
dispersion of particles and reduced Si in the structure of the final composition.

2.8 Temperature Programmed Desorption (TPD) Analysis

The temperature-programmed desorption of ammonia (NH₃-TPD) analysis to investigate the acidic properties and distinguish acid strength and the amounts of acid sites of 8 samples of S₁ - S₈ are shown in Table 5. These samples were selected for NH₃-TPD analysis to provide comparative conditions for the study of time and temperature changes. As previously reported, the NH₃-TPD profiles of SAPO-34 catalysts showed two maxima appeared at about 200 and 400 °C, corresponding to weak and strong acid sites, respectively. The first peak in the NH₃-TPD diagrams is generally related to the weak acidic sites, caused by T-O-H bonds (T can be Al, Si, or P). It is at the center of four-dimensional structures that are only capable of converting methanol to dimethyl ether and are not capable of converting dimethyl ether to light olefins [105–112]. The large area under this peak indicates the number of acidic sites and is proportional to the amount of ammonia excreted, which is higher for the sample S₇ than for the other two samples.

2.9 Catalyst Performance

In Fig. 5(s1-s9), light olefins selectivity of 9 samples in MTO process is shown as a function of reaction time on stream (TOS).

The reaction of methanol conversion over a SAPO-34 catalyst is appeared to include three stages: (a) the dehydration of methanol to DME, (b) the dehydration of DME to olefins and (c) converting olefins to aromatics and paraffin's. Different SiO₂/Al₂O₃ and H₂O/Al₂O₃ molar ratios affect physical and acidic properties of catalysts. Catalysts with various crystallinity, particle size, surface area and acid content show distinct performance in MTO process as displayed in Fig. 5(s1-s9). Low concentration of methanol in the feed beside narrow pore structure of SAPO-34 catalysts leads to gradual diffusion of methanol in active sites. When methanol molecules achieve active sites in SAPO-34 catalysts, products are formed and diffuse out of pore structure [16–35, 113–119]. The crystal size and external surface area of catalysts can help easy admission to active sites too. Samples S1 and S3 with relatively large crystal size and low surface area had long induction time in comparison with other samples. The selectivity of total light olefins reaches to maximum amount when methanol molecules achieve the active sites of catalysts. Catalysts with small crystal size and proper crystallinity showed high total light olefins selectivity according to literatures. Sample S4 showed high total light olefins selectivity, because of its relatively high crystallinity (76%) and small size of crystals (1.3 mm). The maximum ethylene and propylene selectivity for this sample are 50.25 and 21.20, respectively. The selectivity to ethylene is higher than that of propylene for all samples that can be related to propylene and butylene transformation. Propylene and butylene are more reactive than ethylene and transform more easily into oligomers and bigger molecules [120–127]. They might be entrapped in the pores of SAPO-34 samples and thereby could be cracked into ethylene. The cracking of oligomers is reported at around 300 8 C. Conversion of small molecules to larger ones and coke on the surface of catalyst through the secondary reactions leads to catalyst deactivation and prevents product diffusion out of catalyst. Sample S5 with SAPO-34 and SAPO-5 phases was deactivated faster rather than the other catalysts. SAPO-5 is a relative large pore molecular sieve with AFI topology where 1-D straight channel contains the 7.3 A⁺ * 7.3 A⁺ pore entrance and big product molecules can be easily escaped from pores of SAPO-5 without more reaction. Therefore, producing large molecules over sample 5 with SAPO-5 is higher than over SAPO-34 catalyst and blockage of pores.

Table 5 Acidity results determined with NH3-TPD

| Sample no | Acid amount (mmol g⁻¹) | Max peak temperature |
|-----------|------------------------|---------------------|
|           | Weak | Strong | Total acidity | Tₚ₁ | Tₚ₂ |
| S1        | 0.32 | 0.49  | 0.81         | 201 | 419 |
| S2        | 0.31 | 0.55  | 0.87         | 193 | 394 |
| S3        | 0.34 | 0.54  | 0.89         | 189 | 416 |
| S4        | 0.29 | 0.49  | 0.78         | 190 | 400 |
| S5        | 0.49 | 0.88  | 1.37         | 201 | 420 |
| S6        | 0.34 | 0.60  | 0.94         | 199 | 390 |
| S7        | 0.28 | 0.50  | 0.78         | 202 | 388 |
| S8        | 0.36 | 0.38  | 0.74         | 195 | 359 |
by coke production can lead to decrease in lifetime of the catalyst. The acid sites content and crystal size of catalysts are two main factors in deactivation rate of SAPO-34 catalyst. At SiO₂/Al₂O₃ = 0.35, with increasing H₂O/Al₂O₃ molar ratio the crystal size of catalysts increases as mentioned in Table 1. It is expected that the deactivation rate
Fig. 5 (continued)
increases but sample S6 with larger crystals was deactivated slower than S7 with smaller crystal size [128–134]. This is related to low strong acid content in sample S6 that reduces the deactivation rate of catalyst [135, 136]. Mild
acid content for catalyst S6 caused long life time because of decreasing in transformation of short chain olefins to higher molecular weight compounds, which can deactivate the catalyst. Sample S7 with strong and high density of acid sites related to its high Si incorporation, deactivated rapidly. High density of strong acid sites helps hydrogen transfer reaction of olefins to saturated hydrocarbons and aromatics and leads to coke formation on the surface of the catalyst. The large crystal size can also bring diffusion limitation for reactants and products especially in small pore molecular sieves, which may cause coke formation and following deactivation of the catalyst. Sample S6 with small crystal size (1.6 mm) and medium surface area and strong acid site concentration had high total light olefins selectivity and lifetime at investigated time. The smaller crystal size and high external surface area avoided the formation of heavier hydrocarbons because of quick product exit. Sample S8 with low crystallinity (47%) and large crystal size (2.3 mm) because of its high water content had a long intracrystalline diffusion length enough for converting the reaction intermediates to coke which consequently deactivate the catalyst and decrease the light olefins selectivity. The catalytic performances of prepared SAPO-34 samples at 410 °C and 30 wt% methanol in water were investigated in MTO process. For all catalysts, methanol conversion after the first test and before 200 min was obtained over 95%. Catalysts S4, S6 and S7 showed complete conversion. Methanol conversion over time on stream for catalysts S6, S7 and S8 is displayed in Fig. 6. Small crystal size, high acid site content and proper surface area for achieving these active sites can increase methanol conversion.

3 Conclusions

SAPO-34 catalysts were synthesized with 9 different ratios of SiO$_2$/Al$_2$O$_3$ and H$_2$O/Al$_2$O$_3$ under hydrothermal conditions. The morphology of samples was the similar cubic shape of typical SAPO-34. It was indicated that the low ratio of SiO$_2$/Al$_2$O$_3$ in the synthesis gel led to impurity and a high concentration of strong acid sites and an increase of water in the gel mixture led to the incomplete structure of the catalyst. Samples prepared with ratios SiO$_2$/Al$_2$O$_3$ = 0.35, H$_2$O/Al$_2$O$_3$ = 120, and SiO$_2$/Al$_2$O$_3$ = 0.35, H$_2$O/Al$_2$O$_3$ = 50 show less lifetime than other catalysts due to having more and stronger acidic places. The sample S5 prepared with SiO$_2$/Al$_2$O$_3$ = 0.17, H$_2$O/Al$_2$O$_3$ = 60.25. Thus, low water and Si in the catalyst compound lead to the production of SAPO-5 along with SAPO-34 which reduces the light olefin and inactivates the catalyst faster.

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Declarations

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Consent for Publication Not applicable

Conflict of Interest The authors declare that they have no conflicts of interest.

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