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Synthesis of MoO_3 Solid Acid Catalysts by Solution Combustion Approach and Their Catalytic Performance for the Acetalization of Formaldehyde

Yong Gwon Pak[1], Jong Hyok Kim[2,*], Ryong Wan Ham[2], Jun Ho Kim[2], Phyong Mu Pak[1]

Abstract

MoO_3 nanoparticles were synthesized via solution combustion approach. Ammonium nitrate was used as oxidant and ammonium molybdate as fuel in combustion reaction. XRD, FTIR and Raman spectroscopy results showed that MoO_3 nanoparticles were successfully synthesized by the solution combustion reaction. The SEM analysis showed that synthesized MoO_3 nanoparticles have rod-like shape with width and thickness of 50–200 nm and length of 0.2–1 μm, respectively. The XRD pattern and morphology of MoO_3 nanoparticles synthesized by solution combustion method were consistent with those of MoO_3 nanoparticles synthesized by hydrothermal method. This result indicates that MoO_3 nanoparticles with high crystallinity as molybdenum trioxide synthesized by hydrothermal method were synthesized by solution combustion method. MoO_3 nanoparticles synthesized by solution combustion method had higher specific surface area and Brønsted acidity than molybdenum trioxid prepared by thermal decomposition of ammonium molybdate. The molybdenum trioxid acted as solid acid catalyst for the acetalization reaction of formaldehyde with ethanol. Formaldehyde conversion over MoO_3 synthesized by solution combustion method was higher than that over MoO_3 prepared by thermal decomposition of ammonium molybdate. Under batch condition with catalyst concentration of 4% and a reaction time of 90 min, the formaldehyde conversion was achieved up to 44%. This catalyst also exhibited high stability, even after the 8th reaction cycle, the formaldehyde conversion did not decrease significantly.

Keywords: Solution combustion, molybdenum trioxide, ethylal, diethoxymethane, solid acid

INTRODUCTION

The important organic chemical reactions, including isomerization, alkylation, and esterification, use acids as catalysts. The use of solid acids as an alternative to the conventional liquid acid catalysts for these reactions is widely studied. Unlike liquid acids, solid acid catalysts are easy to separate from reaction mixture, little corrosion of equipment, environmentally friendly and little pollution and waste.

Molybdenum trioxid is a widely used solid acid catalyst. MoO_3 has been used as a catalyst for many organic chemical reactions, including esterification [1–4], transesterification [5–7], nitration [8–12], acetalization of glycerol [13, 14], etc. Generally, molybdenum trioxide is supported on various carriers to improve the catalytic activity, selectivity, lifetime and mechanical strength. The supported

***Author for Correspondence**
Jong Hyok Kim
E-mail: jh.Kim1030@star-co.net.kp

[1]Associate Professor, Department of Chemistry, High-Tech Research and Development Center, Kim Il Sung University, South Korea
[2]Professor, Department of Chemistry, High-Tech Research and Development Center, Kim Il Sung University, South Korea

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MoO_3 catalysts have been synthesized by incipient wetness impregnation [1, 15–18], sol-gel method [2, 19, 20], and solid-state method [21, 22]. In particular, the incipient wetness impregnation method is widely used, by which MoO_3 is supported on SnO_2, TiO_2, Al_2O_3, SiO_2 supports.

Like other catalysts, the properties of MoO_3 solid acid catalysts are also affected by the preparation method and the preparation conditions. Huifeng *et al.* found that the hydrodesulfurization activity of MoO_3/Al_2O_3 catalyst was affected by the preparation method [23]. Bernardo *et al*. reported that the catalytic activity for transesterification varied with the calcination temperature of MoO_3 [7]. The variation of catalytic performance with preparation methods and conditions is due to the variation of surface properties and dispersibilites on the support depending on the preparation methods and conditions.

Generally, molybdenum trioxid catalysts are synthesized by precipitation and hydrothermal methods. In addition, the incipient wetness impregnation method is widely used for the preparation of supported catalysts, which involves the formation of molybdenum oxide phase by pyrolysis followed impregnation of ammonium molybdate on the supports [1, 15–18].

The solution combustion method is a very economical method to synthesize nanopowders with high specific surface area, easily and rapidly, with simple equipment. The use of solution combustion method also enables efficient synthesis of supported catalysts [24–26]. However, there is a lack of literature considering the synthesis of MoO_3 solid acid catalysts using solution combustion method and their applications.

On the other hand, the acetalization reaction of formaldehyde is a typical acid-catalyzed reaction and an important organic chemical reaction. For example, diethoxymethane (DEM), a product of the acetalization reaction of formaldehyde with ethanol, has been used as an important organic reaction reagent and as an effective organic solvent [27–29]. However, there is a lack of research using molybdenum oxide catalyst for the acetalization of formaldehyde.

Hence, we have investigated the synthesis of MoO_3 solid acid catalysts by solution combustion approach and their catalytic performance for the acetalization reaction of formaldehyde with ethanol. For comparison, MoO_3 was prepared by pyrolysis of ammonium molybdate and was investigated with the catalyst prepared by solution combustion method.

EXPERIMENTAL

All the reagents viz., molybdenum trioxide, aqueous ammonia solution (25%), ammonium nitrate, ethanol (95%), and formalin (36%) were of analytical grade.

The method of preparing MoO_3 particles by solution combustion method is as follows: Ammonium molybdate ($(NH_4)_2MoO_4$) was used as the fuel and Mo source, and ammonium nitrate (NH_4NO_3) as the oxidant. 7.2 g of MoO_3 was dissolved in the desired amount of ammonia water at 60°C under stirring to prepare an ammonium molybdate solution. 12 g of ammonium nitrate was dissolved in a suitable amount of distilled water and mixed with an ammonium molybdate solution. The quartz beaker with this mixed solution was heated using a hot plate kept at 500°C. Once the majority of the water was evaporated, instantaneous combustion occurred with generation of a large amount of smoke, resulting in formation of grey fine powders.

The method of preparing MoO_3 particles by pyrolysis is as follows: The prepared ammonium molybdate solution was heated to 100°C to evaporate the water and dried. The dried powders were calcined in a muffle furnace at 550°C for 4 h. As a result, blue-white powders were obtained.

XRD patterns of samples were collected with X-ray diffractometer RINT-2000 using Cu Kα (λ=1.54056 Å) according to the Joint Committee on Powder Diffraction Standards (JCPDS) card.

Raman spectroscopy was performed with Raman spectrometer LRS-5 using a green laser (λ=532 nm) as an excitation source. Fourier transform infrared spectroscopy (FTIR) was performed using a Nicolet 6700 FTIR spectrometer in a range between 4000 and 400 cm^{-1}. SEM measurements were performed on a JSM-6610A instrument. BET surface areas were obtained on JW-BK apparatus.

Accurate assessment of the acidity of the unsupported solids through NH_3-TPD was difficult due to the low specific surface area of these materials. Thus, the acidity of pure molybdenum trioxide is estimated as the Brønsted acidity determined by titration [5, 7]. The method of determination of the Brønsted acidity of the catalysts by titration is as follows: In a typical process, 20 ml of NaOH (0.1 $mol.l^{-1}$) was stirred with 100 mg material for 3 h. Using phenolphthalein as indicator, a titration of the supernatant with HCl (0.1 $mol.l^{-1}$) was performed. The acidity was determined in mmol of H^+ per gram of catalyst.

The acetalization reaction of formaldehyde with ethanol can be represented by Eq. (1):

$$HCHO+2C_2H_5OH=C_2H_5O\text{-}CH_2\text{-}OC_2H_5+H_2O \quad (1)$$

The experiments were carried out in a 250 ml three-necked round bottom flask fitted with a reflux condenser and a stirrer. The flask was immersed in a constant temperature bath, temperature of which was maintained within ±1 K of the set point. The flask was charged with 0.5 mol of formalin and 1.25 mol of ethanol and the temperature of the thermostat was set at 343 K. The catalyst was added after the desired temperature was attained. This was considered as zero time for the reaction. Samples were withdrawn at specified intervals and analyzed.

The amount of formaldehyde was determined by the sodium sulfite method. Weighed quantity of sample was treated with excess of sodium sulfite, and the liberated sodium hydroxide was titrated with HCl (0.1 $mol.l^{-1}$) using phenolphthalein as indicator. Water, ethanol and DEM were analyzed using gas chromatograph (GC-14B).

The formaldehyde conversion over the catalyst was evaluated by the molar ratio of the consumed formaldehyde to supplied formaldehyde [30–35].

RESULTS AND DISCUSSION

Generally, in solution combustion methods for the preparation of metal oxide nanoparticles, nitrate of the corresponding metal is used as a metal source and oxidant. However, no nitrate of molybdenum is present. The $(NH_4)_2MoO_4$, which is the most widely used raw material for the synthesis of MoO_3 nanoparticles, has a reducing valency of +6. It contains H and N elements that can be burned to release a large amount of heat and gas in its composition, and it produces NH_3 which can be ignited with nitrogen oxides upon heating. These are some important criteria for fuel in solution combustion reactions. Therefore, ammonium molybdate can be used as Mo source and fuel in solution combustion reaction to synthesize MoO_3 nanoparticles. In this case, ammonium nitrate can be selected as oxidant. The combustion reaction between ammonium nitrate and ammonium molybdate can be represented by Eq. (2):

$$3NH_4NO_3+(NH_4)_2MoO_4=MoO_3+4N_2+10H_2O \quad (2)$$

XRD, FTIR and Raman spectroscopy results showed that MoO_3 nanoparticles were successfully synthesized by the solution combustion reaction.

Figure 1 shows the XRD pattern of the synthesized MoO_3 nanoparticles. The obtained patterns are well matched with standard data file of α-MoO_3(JCPDS:05-0508). It has been confirmed that the MoO_3 nanoparticles are having orthorhombic crystal structure. However, the peak intensities of the powders prepared by solution combustion method are different from those of the powders prepared by pyrolysis of ammonium molybdate. The peak intensities of (0 2 1) and (1 1 0) planes are high for the powders

prepared by pyrolysis method, whereas the peak intensities of (0 k 0) planes including (0 4 0) plane are very high for the powders prepared by solution combustion method. This indicates that MoO_3 nanoparticles synthesized by solution combustion method have high anisotropy. Generally, the MoO_3 nanoparticles synthesized by the precipitation method exhibit XRD patterns, while those prepared by the hydrothermal method also show XRD patterns. In hydrothermal method, MoO_3 nanoparticles with high crystallinity are obtained because molybdic acid is hydrothermally treated at 200°C for more than 12 h [30–35]. Using solution combustion method, MoO_3 nanoparticles with high crystallinity as in hydrothermal method can be easily obtained for a very short time.

The Raman spectra of the sample synthesized by solution combustion method is shown in Figure 2. Raman spectroscopy also confirms that the particles synthesized by solution combustion method are α-MoO_3. The Raman peak at 995 cm^{-1} is assigned to the stretching mode of terminal oxygen (Mo=O). The presence of Raman peak at 820 cm^{-1} indicates the stretching mode of doubly coordinated oxygen (Mo-O-Mo). The peak at 665 cm^{-1} corresponds to the stretching mode of triply coordinated oxygen (Mo_3-O), where the edge-shared oxygens are in common to three MoO_6 octahedra.

These peaks are considered as the fingerprints of the α-MoO_3 phase. The bending and scissoring modes of Mo_3–O are observed at 337 and 378 cm^{-1}, respectively. The peak at 287 cm^{-1} is related to double bond O=Mo=O and the peak at 168 cm^{-1} is related to lattice vibrations [34, 35]. The Raman spectra of MoO_3 prepared by pyrolysis showed the same shape as that of MoO_3 synthesized by solution combustion method.

The FTIR spectra of the sample synthesized by solution combustion method are shown in Figure 3. The peak at 990 cm^{-1} corresponds to the stretching vibration of terminal oxygen (Mo=O), and the peak at 870 cm^{-1} is related to the stretching vibration of doubly coordinated oxygen (Mo-O-Mo). And the peak at 560 cm^{-1} is due to the stretching vibration of oxygen coordinated to three Mo atoms (Mo_3-O) [7, 32]. Thus, the FTIR spectra also confirms the formation of α-MoO_3 by solution combustion method. The FTIR spectra of the sample prepared by pyrolysis method also showed the same shape as that of MoO_3 synthesized by solution combustion method.

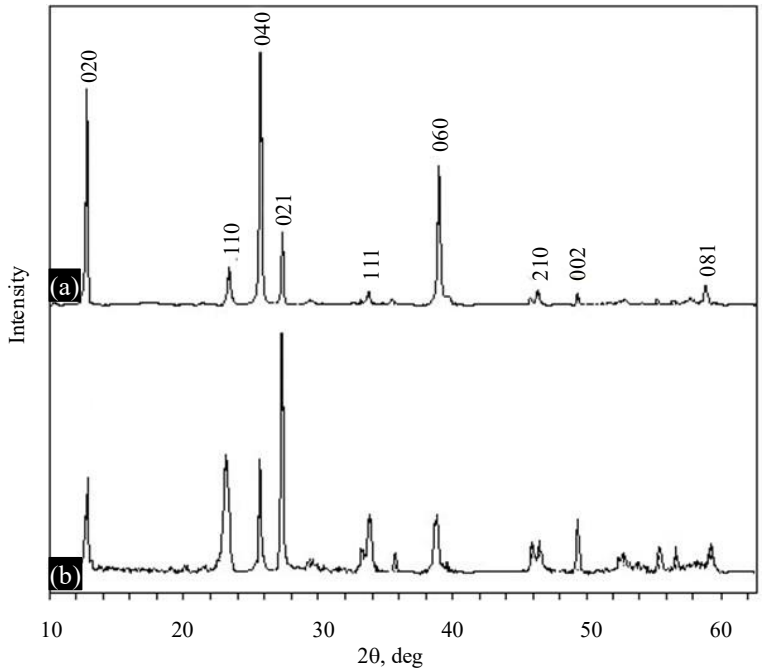

Figure 1. (a, b) XRD patterns of MoO_3 particles synthesized by (a) solution combustion method, and (b) pyrolysis.

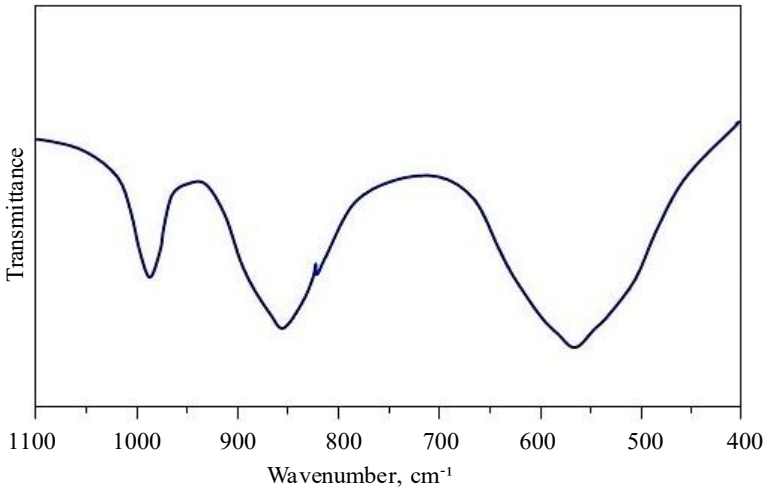

Figure 2. Raman spectra of MoO_3 nanoparticles synthesized by solution combustion method.

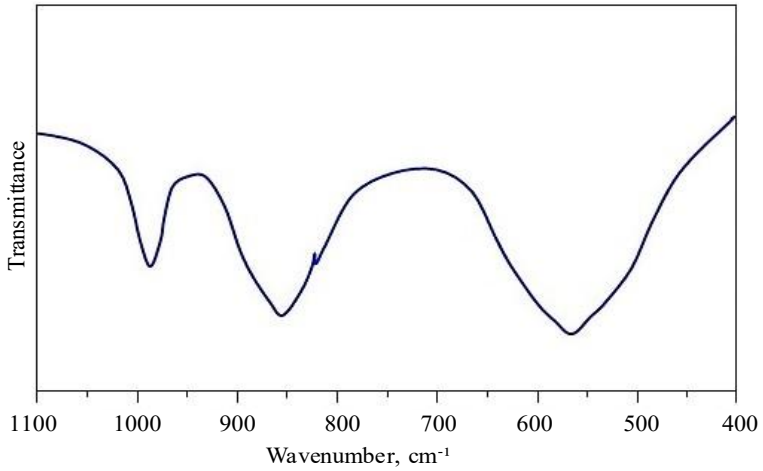

Figure 3. FTIR spectra of MoO_3 nanoparticles synthesized by solution combustion method.

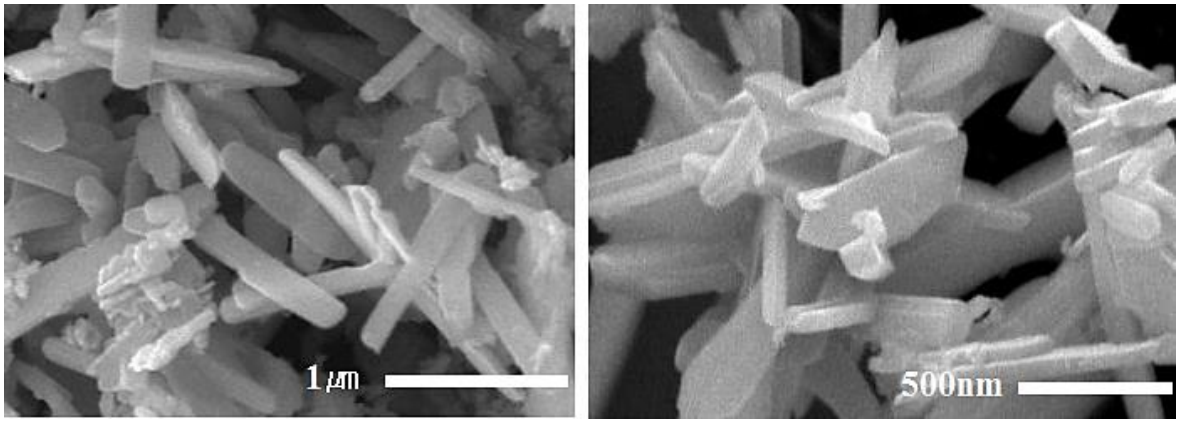

Figure 4. SEM image of MoO_3 nanoparticles synthesized by solution combustion method.

Figure 4 shows the SEM micrograph images of MoO_3 nanoparticles synthesized by solution combustion method. It reveals that the MoO_3 nanoparticles have roughly uneven size with width and thickness of 50–200 nm, and length of 0.2–1 μm.

MoO_3 nanoparticles synthesized by pyrolysis or precipitation methods have hexagonal plate-like structure, and the MoO_3 nanoparticles synthesized by hydrothermal method have rod-like structure [30–

35]. MoO_3 nanoparticles synthesized by solution combustion method also have rod-like structure as MoO_3 nanoparticles synthesized by hydrothermal method. This is in good agreement with the XRD results that MoO_3 nanoparticles synthesized by solution combustion method have high anisotropy.

The specific surface area and Brønsted acidity of MoO_3 nanoparticles synthesized by solution combustion method are given in Table 1. For comparison, the properties of molybdenum trioxide synthesized by the pyrolysis of ammonium molybdate are also presented. It can be seen that the specific surface area of MoO_3 synthesized by solution combustion method is higher than that of MoO_3 synthesized by pyrolysis method. In pyrolysis method, the decomposition of ammonium molybdate during calcination produces MoO_3, whereas in solution combustion method, MoO_3 is produced by the combustion reaction between ammonium nitrate and ammonium molybdate. During the combustion reaction, a large amount of heat and gases are generated, which leads to the instantaneous formation of powders with small size and high specific surface area. Due to the higher specific surface area, the acidity of MoO_3 synthesized by solution combustion method is higher than that of MoO_3 synthesized by pyrolysis method.

On the other hand, in literature, the MoO_3 precursors synthesized by hydrothermal method were calcined at different temperatures and their Brønsted acidity was measured, and the results showed that the acidity of the sample calcined at 600°C was the highest, with a value of 16.2 $mmol.g^{-1}$ [7]. The acidity of MoO_3 synthesized by solution combustion method is higher than that.

MoO_3 acts as Brønsted acid in acid-catalyzed reactions, and the acid sites are hydroxyl groups formed on the surface (Mo-OH or Mo-(OH)-Mo). MoO_3 can also act as a Brønsted acid in the acetalization reaction of formaldehyde with ethanol.

Figure 5 shows the evolution of the formaldehyde conversion with reaction time over MoO_3 catalysts. The catalyst concentration was 5 wt% of the total reactant. As can be seen, the formaldehyde conversion over the catalyst prepared by solution combustion method is higher than that over the catalyst prepared by pyrolysis method.

Table 1. Specific surface area and Brønsted acidity of MoO_3 samples.

Preparation method	Specific surface area ($m^2\ g^{-1}$)	Brønsted acidity ($mmol\ g^{-1}$)
Solution combustion	11.6	16.8
Pyrolysis	5.3	15.4

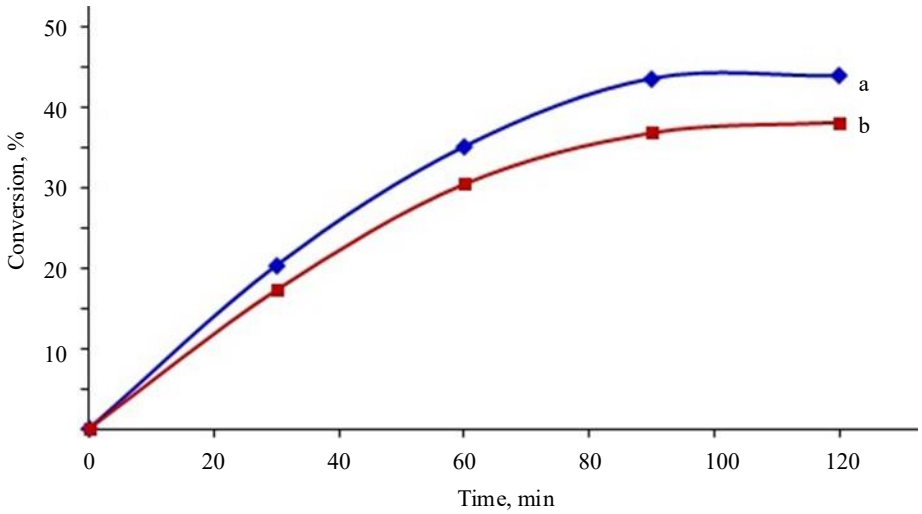

Figure 5. Formaldehyde conversion with reaction time for MoO_3 catalysts synthesized by solution combustion method (a) and pyrolysis.

The reason is that, as discussed earlier, the catalyst prepared by solution combustion method has a higher specific surface area and Brønsted acidity than the catalyst prepared by pyrolysis method.

On the other hand, the (0 2 0), (0 4 0) and (0 6 0) crystal planes in the MoO_3 solid acid catalyst affect the catalytic performance. The MoO_3 precursors synthesized by hydrothermal method were calcined at different temperatures and evaluated for catalytic properties in transesterification reactions, and the results showed that the MoO_3 catalysts with strong XRD peak intensities of these crystal planes have high solid acidity and also provide improved yields [7]. As considered in XRD analysis, the peak intensities of (0 2 0), (0 4 0) and (0 6 0) crystal planes are very high for MoO_3 synthesized by solution combustion method. It can be seen that these crystalline properties also affect the catalytic performance.

Next, the effect of the concentration of MoO_3 solid acid catalyst was investigated. The catalyst concentration was defined as a percentage of the catalyst mass to the total reactant mass. The reaction time was 90 min. The results clearly show that the formaldehyde conversion increased with increasing catalyst concentration (Figure 6). This increase in formaldehyde conversion can be attributed to the increase in the total number of acid sites available for reaction with the increase in the catalyst concentration. And when the catalyst concentration is higher than 4%, the catalyst concentration has little effect on the formaldehyde conversion. As can be seen from the experimental results, the formaldehyde conversion over MoO_3 solid acid catalyst synthesized by solution combustion method is around 44% under batch experimental conditions. This is a high conversion that can be compared to the conversion of the case where cation exchange resin was used as a solid acid catalyst [29].

Recycling experiments were performed to study the stability of the catalyst. For the reusability studies, the reactions were performed under a catalyst concentration of 4% and a reaction time of 90 min. From one reaction to the other, the catalyst was filtered off and used without washing. Figure 7 shows that the catalyst is very stable up to four cycles and it actually presents high catalytic activity up to 8-times, with the potential to be used in further consecutive runs. The reason for the slow decrease in conversion with increasing catalyst utilization is that molybdenum oxide is leached into the medium during the reaction. During the reaction, the medium was changed to blue color because Mo species was dissolved in the medium [2, 5].

MoO_3 nanoparticles were synthesized by solution combustion method and their solid acid catalytic performance was investigated for the acetalization reaction of formaldehyde. Scheme of the synthesis of MoO_3 nanoparticles by solution combustion method and their solid acid catalysis are given in Figure 8. First, MoO_3 nanoparticles were synthesized by solution combustion method. Ammonium nitrate was used as an oxidant and ammonium molybdate as fuel in combustion reaction. XRD, FTIR and Raman spectroscopy results showed that MoO_3 nanoparticles were successfully synthesized by the solution combustion reaction.

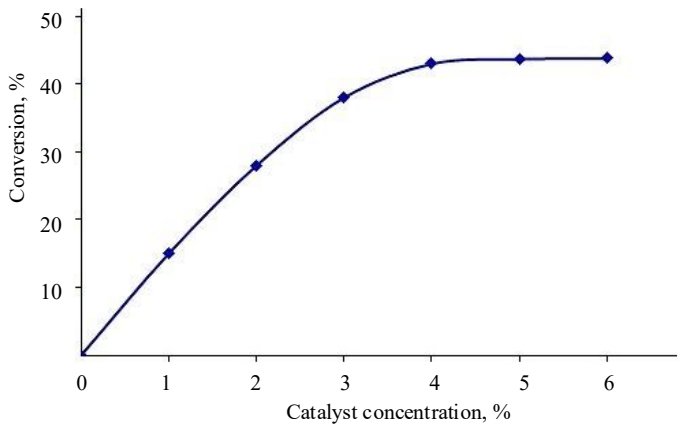

Figure 6. Formaldehyde conversion with catalyst concentration.

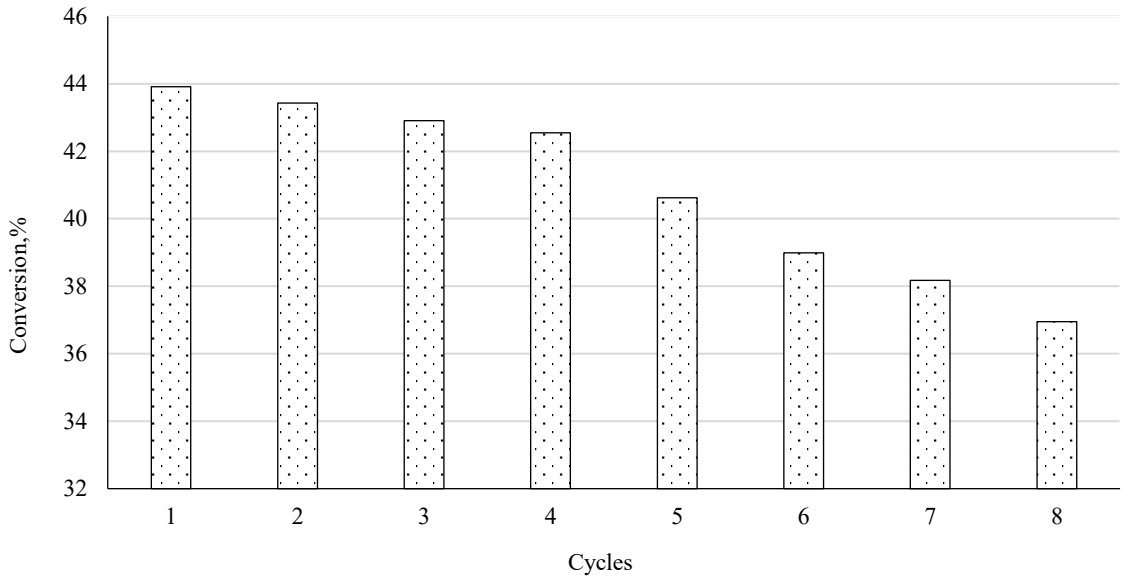

Figure 7. Reusability of the catalyst.

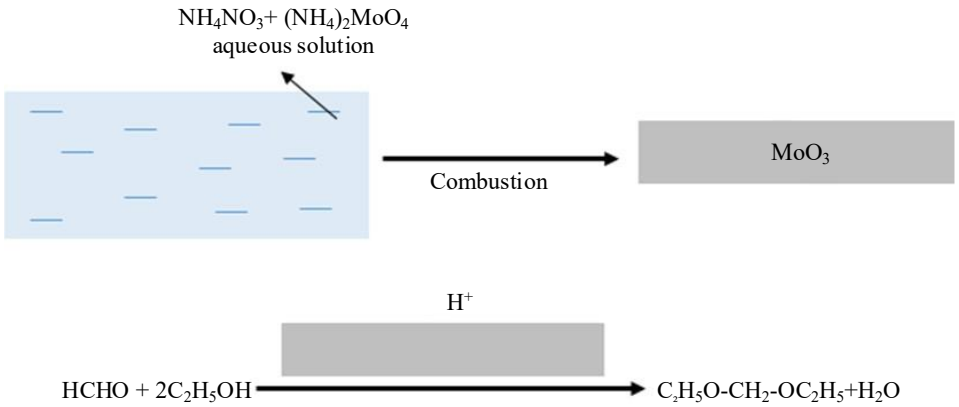

Figure 8. Scheme of the synthesis of MoO_3 by solution combustion method and their solid acid catalysis.

CONCLUSION

The SEM analysis showed that synthesized MoO_3 nanoparticles have rod-like shape with width and thickness of 50–200 nm and length of 0.2–1 μm. Next, the solid acid catalytic performance of MoO_3 nanoparticles synthesized by solution combustion method was investigated. It exhibited good solid acid catalytic performance for the acetalization reaction of formaldehyde with ethanol. Under batch condition with catalyst concentration of 4% and a reaction time of 90 min, the formaldehyde conversion was achieved up to 44%. This catalyst also exhibited high stability, even after the 8th reaction cycle, the formaldehyde conversion was not decreased significantly. MoO_3 synthesized by solution combustion method had higher specific surface area and Brønsted acidity than MoO_3 synthesized by thermal decomposition of ammonium molybdate, and thus exhibited better catalytic performance for the acetalization reaction of formaldehyde with ethanol. The results indicate that the solution combustion method can be more effective than the incipient wetness impregnation method when preparing supported catalysts.

Highlights

1. MoO_3 nanoparticles can be synthesized by solution combustion reaction using ammonium nitrate as oxidant and ammonium molybdate as fuel.
2. MoO_3 nanoparticles synthesized by solution combustion method have excellent solid acid catalytic performance for the acetalization reaction of formaldehyde with ethanol.

3. The solution combustion method can be more effective than the incipient wetness impregnation method when preparing supported catalysts.

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