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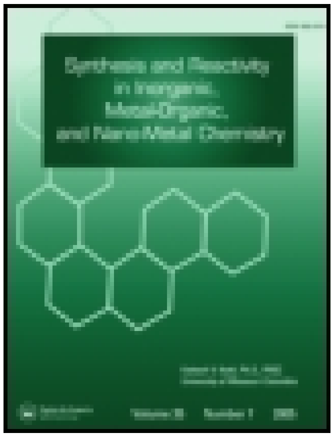

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Preparation of 2,3-diarylthiazolidin-4-one derivatives using Barium molybdate nano powders

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Abstract

Barium molybdate ($BaMoO_4$) nano-powder was successfully synthesized *via* a simple precipitation route without any surfactant. The prepared nano-powder was applied for the facile synthesis of 2,3-diarylthiazolidin-4-one derivatives using cyclization reaction of aromatic aldehydes, aromatic amines and thioglycolic acid under ambient condition.

Keywords: thioglycolic acid; thiazolidin-4-one; Barium molybdate ($BaMoO_4$); 2,3-diarylthiazolidin-4-one

Introduction

Barium molybdate ($BaMoO_4$) has attracted much attention due to their unique structure and potential applications. The more attraction of barium molybdate is due to its attractive photoluminescence (PL) property [1-7]. $BaMoO_4$ existing in the scheelite structure, has a high potential for applications in many fields, such as optical fibers, light-emitting diodes, lanthanide activated lasers, and scintillation detectors [1-7]. Various techniques for the preparation of $BaMoO_4$ were reported including Czochralski technique [6], spontaneous crystallization method [8] Complex Polymerization Method (CPM) [9], simple precipitation method [10], reverse microemulsions [11], microwave-assisted hydrothermal [12], EDTA-mediated route [13] and calcinations method [14]. However, the preparation and evaluation of the catalytic properties of $BaMoO_4$ in organic transformations have not been focused. Thus the present work concentrates on the homogeneous precipitation, preparation and application of $BaMoO_4$ nanopowder as an efficient catalyst in the synthesis of a variety of 2,3-diarylthiazolidin-4-one derivatives (Scheme 1).

The heterocycles containing thiazolidin-4-ones are of great interest and have been preclinically shown to be useful because they have associated with a diverse range of interesting biological activities, such as anticancer [15], anti- HIV [16], antimalarial [17], tuberculostatic [18], antihistaminic [19], anticonvulsunt [20], antibacterial [21], and antiarrythmic [22].

Therefore, construction of new methods for the preparation of thiazolidin-4-ones still holds great interest. A variety of synthetic methods have been developed for constructing such thiazolidin-4-

ones and among them the cyclo-condensation reaction of aldehydes, amines and thioglycolic acid has been more versatile [23-32].

Based on the above information's and due to our interest in developing the synthetic methodologies for the construction of heterocyclic compounds [33-38], herein, a detailed account of our focused attention toward construction of the thiazolidin-4-one derivatives was reported, *via* a cyclo-condensation reaction of aldehydes, amines and mercaptoacetic acid in the presence of $BaMoO_4$ nanopowder as catalyst (Scheme 1).

Experimental

Reagents and Instrumentation:

All reagents were purchased from Merck and Aldrich and used without further purification. All yields refer to isolated products after purification. The NMR spectra were recorded on a Bruker Avance DPX 400 MHz instrument. The spectra were measured in DMSO-d_6 relative to TMS (0.00 ppm). Elemental analysis was performed on a Heraeus CHN-O-Rapid analyzer. TLC was performed on silica gel polygram SIL G/UV 254 plates. The powder X-Ray diffraction patterns were measured with D_8, Advance, Bruker, axs, diffractometer using CuKα irradiation. FE-SEM was taken by a Hitachi S-4160 photograph to examine the shape and size of $BaMoO_4$ nano-particles.

Preparation of $BaMoO_4$ nano-powder: To an aqueous solution of $BaCl_2$ (10 mmol $BaCl_2$ in 50 mL of water) sodium molybdate (10 mmol dissolved in 50 mL of EtOH/H_2O (50/50) + triethyl ammonium chloride (15 mmol) + 5 mL NH_3 (aqueous solution 37%)) was added drop-wise with vigorously stirring at room temperature. After the dropping process was completed, the resultant mixture was aged for 30 min. The accessed precipitates were separated from the mother liquor by filtrating and were washed with distilled water for several times, then were dried at 100 °C. The $BaMoO_4$ nano-particles were slightly ground for analysis.

General procedure: To a mixture of aldehyde (1 mmol), aromatic amine (1 mmol) and thioglycolic acid (1 mmol) in ethanol (5 mL), $BaMoO_4$ nano-powder (0.17 mmol) as a catalyst was added and the mixture was stirred for appropriate time at room temperature (Table 2). Progress of the reaction was monitored by TLC. Upon completion, the solvent was concentrated and the reaction mixture was diluted in CH_2Cl_2, the catalyst was isolated by simple filtration, and the crude product was crystallized in *n*-hexane / ethyl acetate to afford the pure product.

Selected data:

2-(4-hydroxyphenyl)-3-p-tolylthiazolidin-4-one (Table 2, Entry 7): ^{1}H-NMR (400 MHz, $CDCl_3$): 2.27 (s, 3H), 3.71 (d, J = 12.4 Hz, 1H), 3.82 (d, J = 12.4 Hz, 1H), 5.14 (s, 1H), 6.70-7.14 (m, 6H), 7.73 (d, J = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 20.6, 34.0, 52.3, 115.7, 120.8, 129.7, 130.8, 148.7, 157.2, 159.3, 160.7, 170.4 ppm; Found: C, 67.36; H, 5.35; N, 5.02 $C_{16}H_{15}NO_2S$; requires: C, 67.34; H, 5.30; N, 4.91%].

Results and discussion

In order to determine the crystalline structure and phase composition of the $BaMoO_4$ nano-powder, X-ray diffraction (XRD) analysis using Cu-Kα radiation was applied (Figure 1). The XRD pattern of $BaMoO_4$ nano-powder exhibited diffraction pattern characteristics of the tetragonal structure with pronounced 26.67, 27.27, 32.52, 34.37, 36.42, 38.52, 43.42, 46.32, 48.27, 53.47, 54.62, 67.62 and 69.62 peaks (Figure 1). The average crystallite diameter of the as prepared powder was determined by XRD, using from the highest peak of the $BaMoO_4$ phase (2θ at around 26.67), according to the Scherrer equation. The average crystalline size estimated by using the Scherrer formula from the highest peak of the XRD was 80 nm and was determined with the use of the following equation:

Average Crystalline Size :

$$\frac{K\lambda}{\beta \cos\theta}$$

Where *K* is taken as 0.9, λ is the wavelength of X-ray used, β is the full width half maximum of the prominent peak and θ is the glancing angle.

The average crystalline size of the $BaMoO_4$ nano-powder was characterized by field emission scanning electron microscopy (FE-SEM). Figure 2 shows the FE-SEM micrographs of the $BaMoO_4$ nano-particles. Based on the FE-SEM observation, the $BaMoO_4$ nano-particles contain spherical or ellipsoidal shapes. The particle sizes were mainly distributed in the range of 60 to 120 nm. The size of about 50 particles of $BaMoO_4$ was measured and the average particle size was obtained as 83 nm.

Figure 3 shows grain size distributions of $BaMoO_4$ nano-powders. The powder milled for 2h and sonicated in ethanol for1h had monomodal distributions with median particle size.

A comparison between the particle size distributions of $BaMoO_4$ samples measured by different techniques, including Cyclic microwave irradiation, Complex polymerization method (CPM), Precipitation method, Hydrothermal Synthesis using domestic microwave with the present study was done and the results are summarized in Table 1. It is shown that particle size obtained with the present work is comparable with those of reported literature.

As a preliminary test reaction, catalytic conversion of three-component condensation of benzaldehye, 4-methyl aniline and thioglycolic acid to 2-phenyl-3-*p*-tolylthiazolidin-4-one was examined (Scheme 2).

To find optimize the reaction conditions, the reaction was carried out by the using of different solvents (Table 2, Entries 1-7) or solvent-free condition (Table 2, Entry 8). Among the solvent screened, ethanol was found to be the best promoter in terms of reaction times and yields (Table 2, Entry 5).

For find the optimized amount of $BaMoO_4$, the reaction was carried out by varying amount of the catalyst (Table 2, Entries 9-12). Maximum yield was obtained when 0.17 mmol of catalyst was used (Table 2, Entry 5). A further increase in the amount of $BaMoO_4$ nano-powder in mentioned reaction did not have any significant effect on the product yield. Under the standard conditions, no reaction was observed in the absence of a catalyst. This shows that the catalyst is essential for the product formation (Table 2, Entry 13). The results are summarized in Table 2.

Next, the scope and efficiency of this procedure was explored for the synthesis of a wide variety of substituted 2,3-diarylthiazolidin-4-one derivatives (Scheme 1, Table 3).

As expected, this reaction proceeded smoothly and the desired products were obtained in good yields. In general, aromatic aldehydes and aromatic amines were well tolerated in this reaction system (Table 3). Based on the obtained results, the electronic effects and the steric effects of the substituents both in amines and aldehydes played significant role in the yields of products. Aromatic aldehyde systems that possessed substitutions at the *ortho*, *meta* or *para* positions had the yield, however, aromatic aldehydes containing electron-donating groups gave higher yields than that with electron-withdrawing groups. When *ortho*-substituted aldehydes (Table 3, Entry 4) were used in this process, the corresponding product was obtained in good yields. Electron donating group on the amine was able to facilitate the transformation by giving evidently higher yield of products than the entries using halogen atom functionalized anilines (Table 3).

The work-up procedure is very clear-cut; that is the products were isolated and purified by simple filtration and recrystallization from *n*-hexane / ethyl acetate.

In order to estimate the efficiency and generality of this methodology, the obtained results in the synthesis of 2,3-diphenyl thiazolidin-4-one using $BaMoO_4$ nanopowder as catalyst has been compared with those of the previously reported methods. The results are summarized in Table 4. It was found that the present method is convincingly superior to the reported methods with respect to reaction time and the yield of the product.

Our attention was then turned to the possibility of recycling the catalyst from the reaction media since the recovery and reuse of the catalyst are highly preferable for a greener process. At the completion of the reaction, the reaction mixture was poured into $CHCl_3$ and stirred for 5 min. The solid catalyst separated was filtered via simple filtration, dried and reused for subsequent reactions. The reusability of the catalyst was investigated by using benzaldehye, 4-methyl aniline and thioglycolic acid as model substrates. After 10 recycles, the catalyst still had a high activity and gave the corresponding product in good yield (Figure 4).

Conclusion

The nanopowder structure, $BaMoO_4$, prepared by a simple precipitation method, was used as an efficient catalyst for the preparation of 2,3-diarylthiazolidin-4-one derivatives. It was stated that the use of 0.17 moles of $BaMoO_4$ as catalyst for the catalytic condensation reaction of aromatic aldehydes, aromatic amines and thioglycolic acid under ambient condition provides high yields of products. Certainly, the simplicity of the accommodated catalytic system makes it suitable for further research and practical applications.

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Table 1: Particle size distribution of $BaMoO_4$ nano-powder compared with those of reported literatures.

Entry	Method	Particle size	Reference
1	Cyclic microwave irradiation	30-75	4
2	Complex polymerization method (CPM)	40-50 nm	9
3	Precipitation method	31.52 ± 4.65 nm	10
4	Hydrothermal Synthesis using domestic microwave	0.7-3.1 μm	39
5	This work	83 nm	-

Table 2: Optimization of the reaction conditions in the synthesis of 2-phenyl-3-*p*-tolylthiazolidin-4-one (Scheme 2)

Entry	Catalyst (mmol)	T (°C)	Solvent (5 mL)	Yield (%)[a]
1	0.17	r.t.	*n*-Hexane	-
2	0.17	r.t.	CH_2Cl_2	25
3	0.17	r.t.	Et_2O	30
4	0.17	r.t.	EtOAc	51
5	0.17	r.t.	EtOH	88
6	0.17	r.t.	MeOH	85
7	0.17	r.t.	CH_3CN	75
8	0.17	r.t.	-	68
9	0.34	r.t.	EtOH	86
10	0.09	r.t.	EtOH	80
11	0.05	r.t.	EtOH	45
12	0.03	r.t.	EtOH	27
13	-	r.t.	EtOH	-

[a] Isolated Yields, reaction time: 2 h

Table 3: Synthesis of 2,3-diarylthiazolidin-4-one derivatives using $BaMoO_4$ nano-powder as catalyst (Scheme 1)

Entry	Aldehyde	Amine	Time (h)	Yield (%)a
1	Benzaldehyde	4-Methylaniline	2	88
2	4-Methylbenzaldehyde	4-Methylaniline	2	85
3	4-Chlorobenzaldehyde	4-Methylaniline	2.5	74
4	2-Chlorobenzaldehyde	4-Methylaniline	4	68
5	3-Nitrobenzaldehyde	4-Methylaniline	3.5	77
6	4-Methoxybenzaldehyde	4-Methylaniline	2	84
7	4-Hydroxybenzaldehyde	4-Methylaniline	3	71
8	2-Chlorobenzaldehyde	4-Methoxyaniline	4	60
9	4-Nitrobenzaldehyde	4-Methoxyaniline	4	56
10	3-Nitrobenzaldehyde	4-Methoxyaniline	3.5	61
11	2-Methylbenzaldehyde	4-Methoxyaniline	2.5	71

12	4-Methoxybenzaldehyde	4-Methoxyaniline	2	80
13	4-Methylbenzaldehyde	4-Methoxyaniline	2	87
14	Benzaldehyde	4-Chloroaniline	3	57
15	Benzaldehyde	Aniline	2h	81

[a] Isolated yields. All known products have been reported previously in the literature and were characterized by comparison of NMR spectra with authentic samples [25-32].

Table 4: Comparison results of $BaMoO_4$ with other catalysts reported in the literature

Entry	Catalyst	Condition	Yield (%)[a]	Reference
1	$Bi(SCH_2COOH)_3$	Solvent-free; 70 °C; 2h	75	30
2	H_2SO_4	Toluene, 100 °C; 5 h	34	31
3	*P*-TsOH	Toluene, 100 °C; 5 h	32	31
4	MsOH	Toluene, 100 °C; 5 h	24	31
5	$HClO_4$-TiO_2	Toluene, 100 °C; 5 h	48	31
6	Acidic Al_2O_3	Toluene, 100 °C; 5 h	15	31
7	TiO_2	Toluene, 100 °C; 5 h	8	31
8	Amberlite	Toluene, 100 °C; 5 h	36	31
9	Zeolite type Y	Toluene, 100 °C; 5 h	41	31
10	$BaMoO_4$	Ethanol; r.t.; 2 h	81	This work

[a] Isolated Yield; based on the preparation of 2,3-diphenyl thiazolidin-4-one

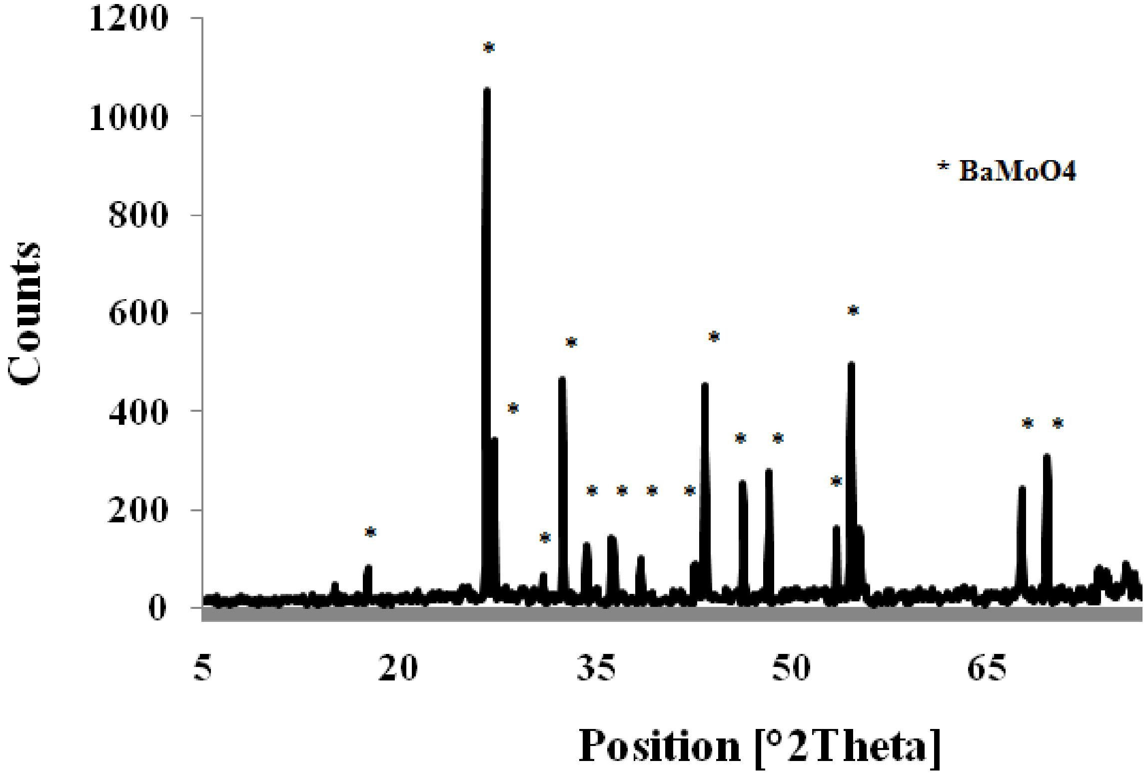
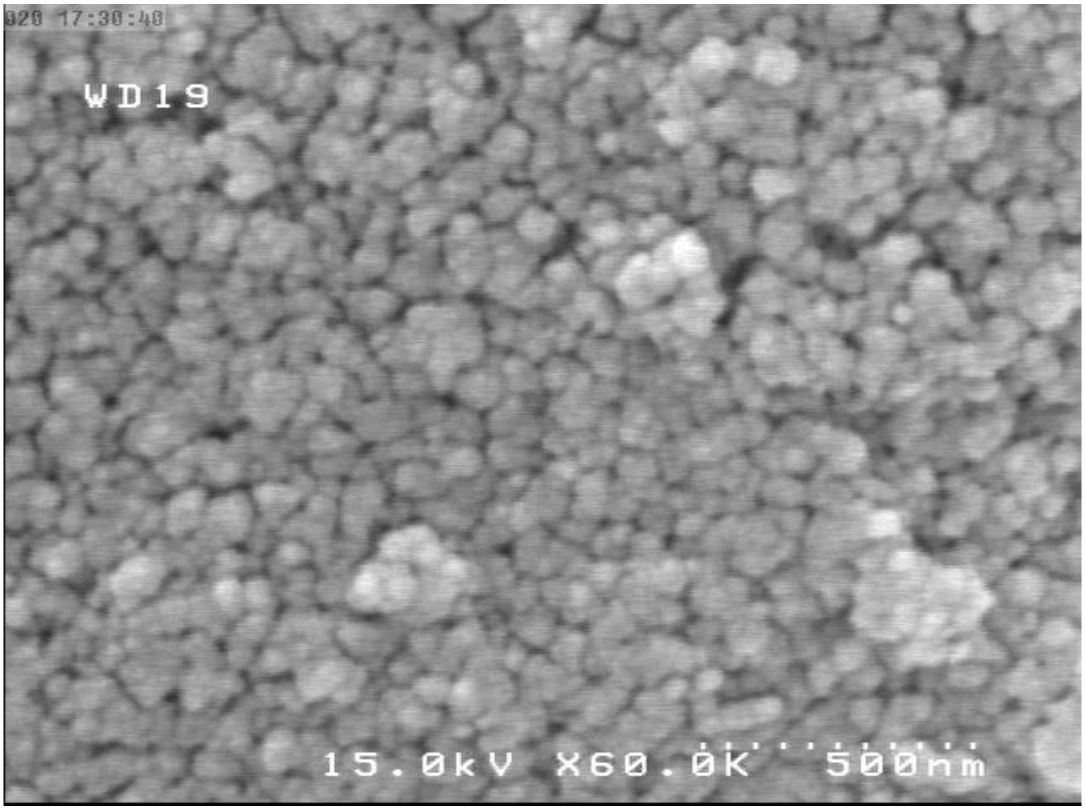


Figure 1: XRD pattern of $BaMoO_4$ nano-particles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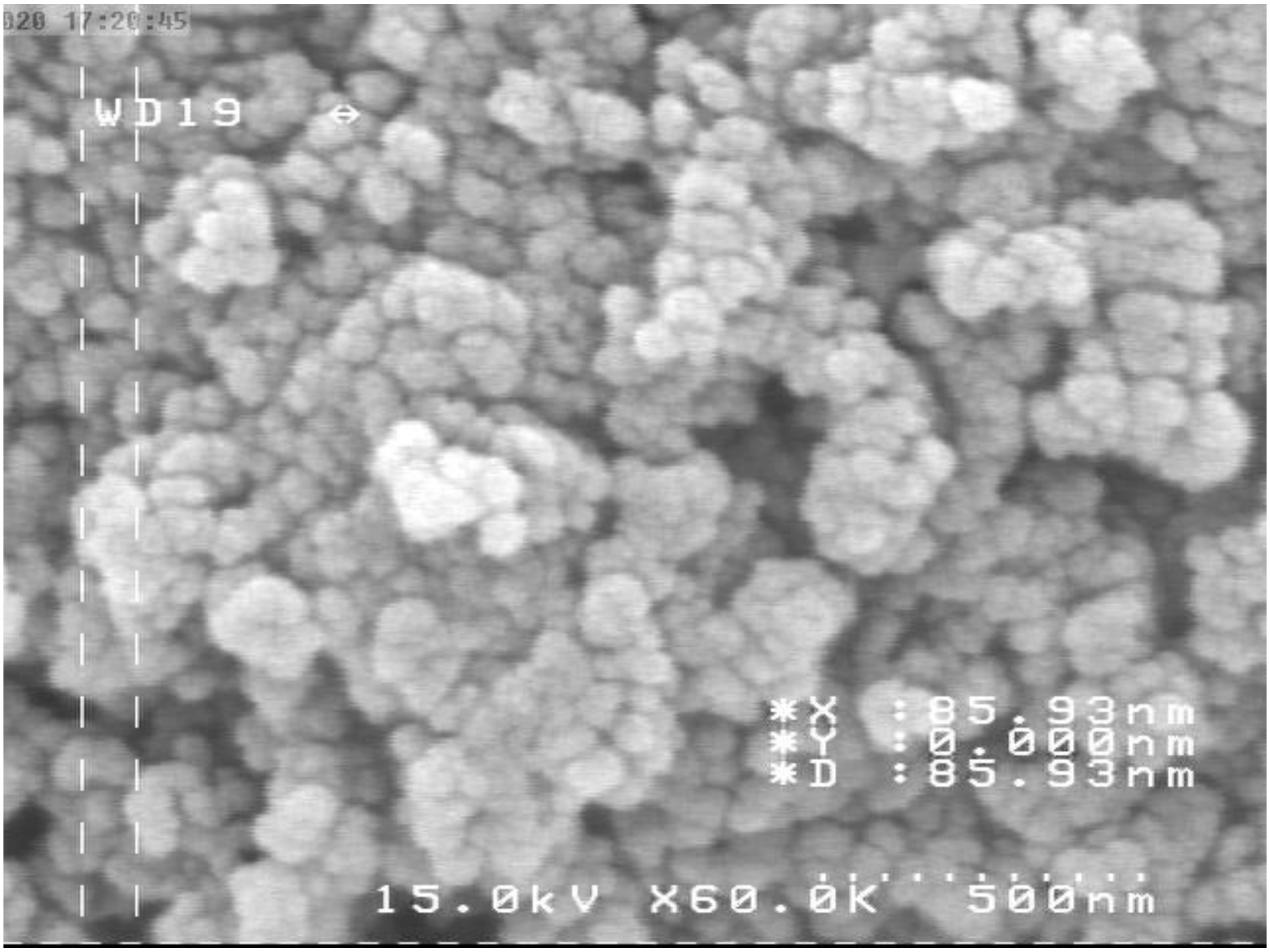
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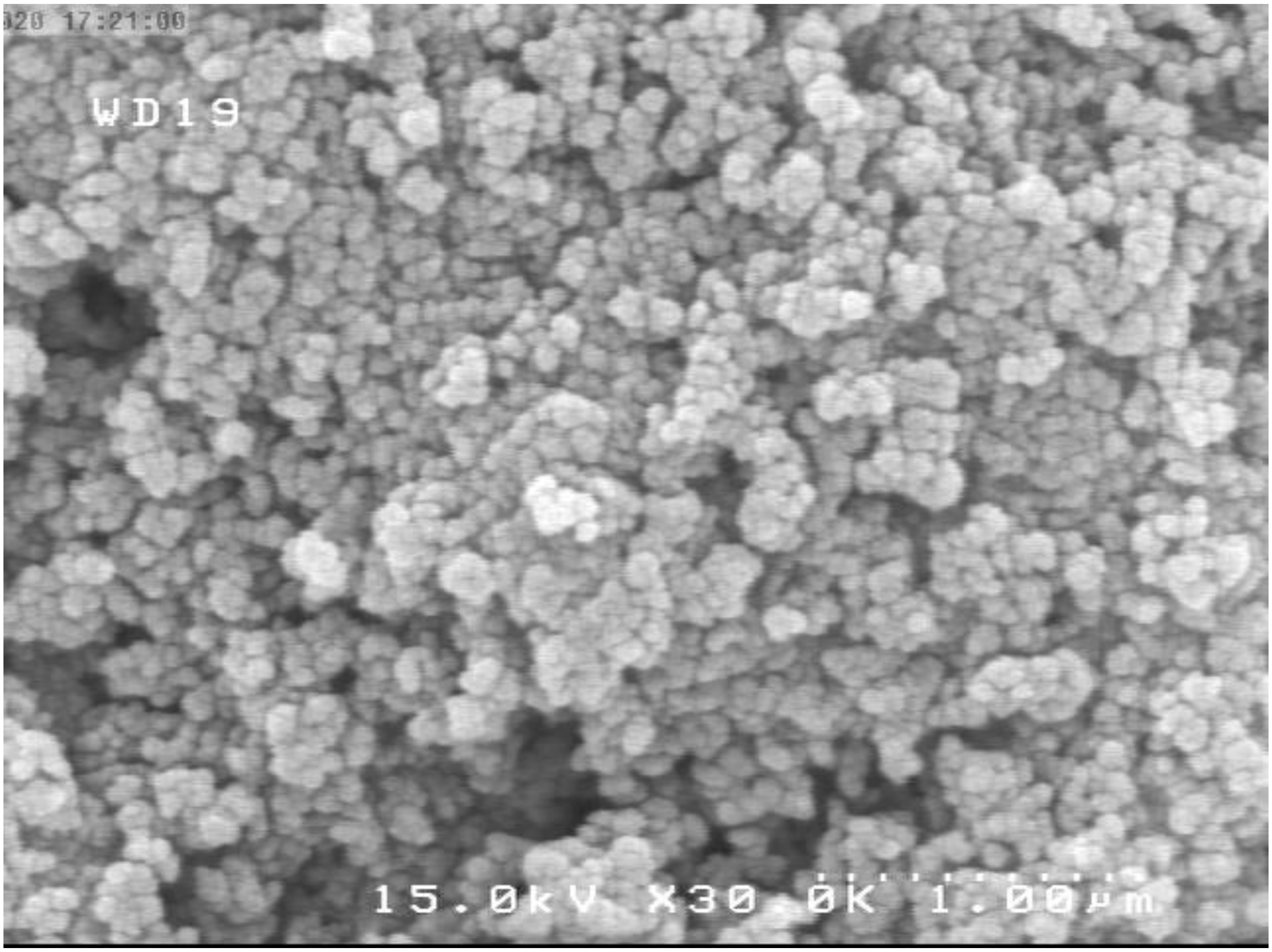


Figure 2: FE-SEM micrographs of $BaMoO_4$ nano-particles

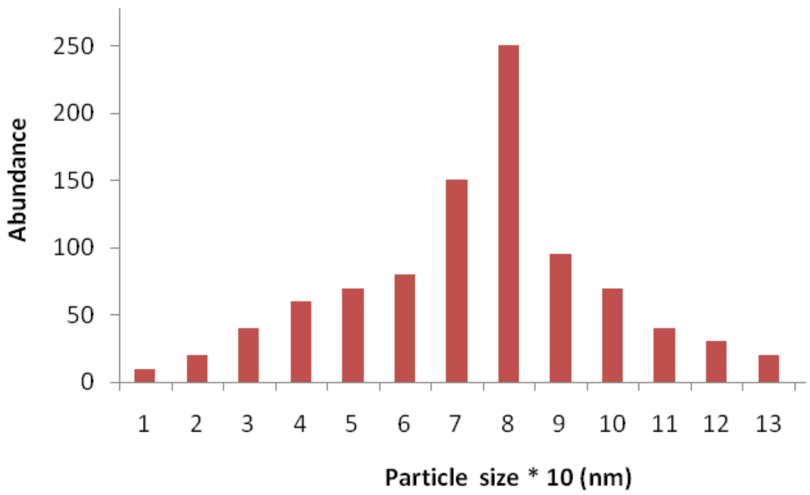


Figure 3: Particle size distribution of $BaMoO_4$ nano-particles

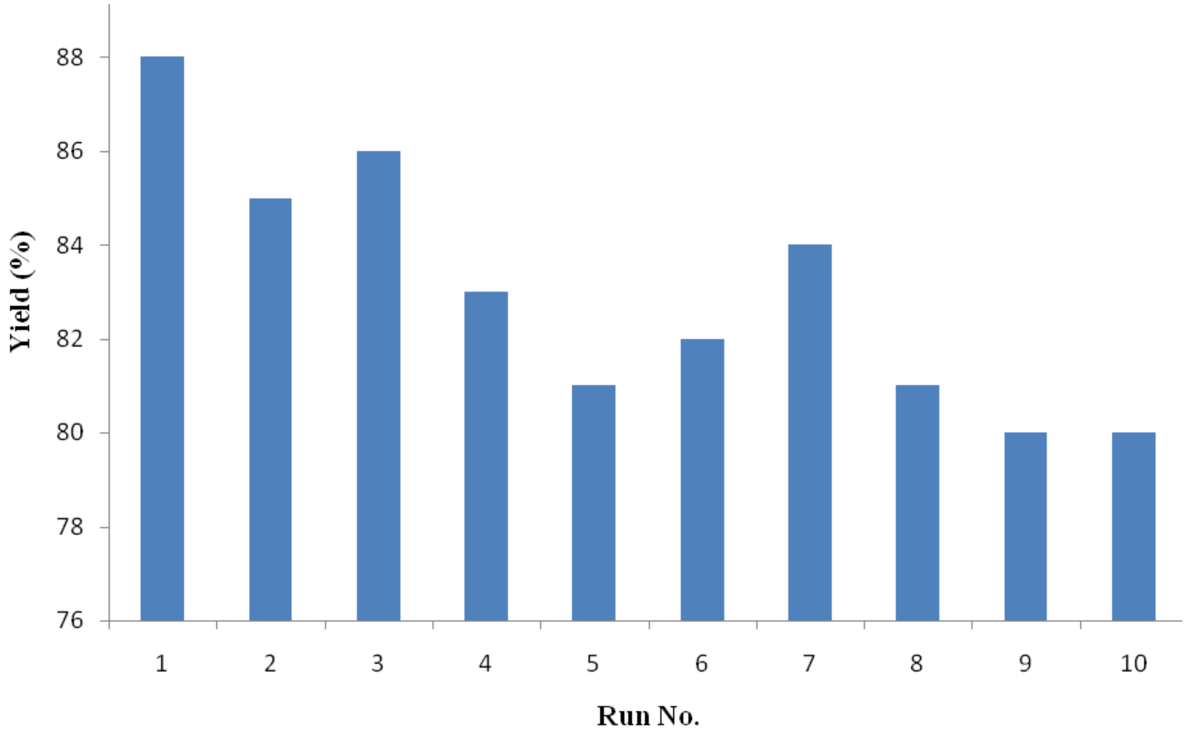
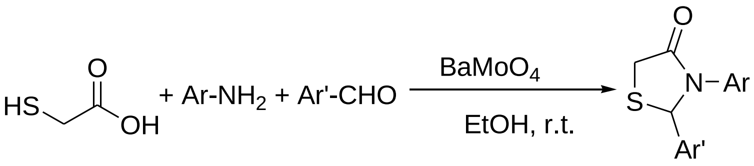
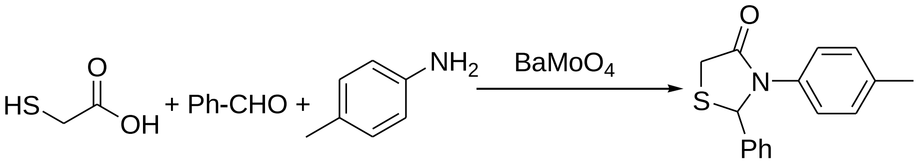


Figure 4: Rusability of $BaMoO_4$ nano-powder



Scheme 1: Preparation of 2,3-diarylthiazolidin-4-one derivatives



Scheme 2: Preparation of 2-phenyl-3-*p*-tolylthiazolidin-4-one