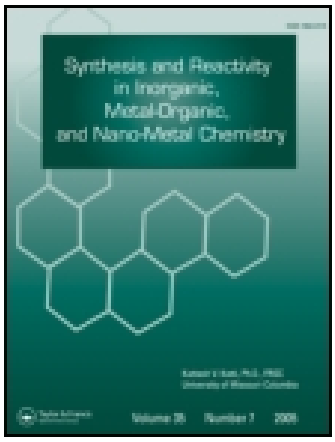

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Synthesis and Characterization of Nanocrystalline In_2O_3 and Its Efficacy as a Catalyst for the One Pot Synthesis of Amidoalkyl Naphthols

Vishvanath D. Patil [a] , Jyotsna S. Thakur [a] , Shramesha Mhatre [a] , Medha Gole [a] & Aarti Jaiswal [a]
[a] Department of Chemistry , C.K. Thakur College , New Panvel(w) , India
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Synthesis and Characterization of Nanocrystalline In_2O_3 and Its Efficacy as a Catalyst for the One Pot Synthesis of Amidoalkyl Naphthols

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Department of Chemistry, C.K. Thakur College, New Panvel(w), India

In_2O_3 nanopaticles were synthesized by glycine-nitrate combustion method. The synthesized In_2O_3 is characterized by the powder X-ray diffraction and transmission electron microscopy. The average crystallite size, as determined by Scherrer's formula, was found to be about 48 nm. A catalytic amount of In_2O_3 nanopaticles, were used for an efficient synthesis of amidoalkyl naphthols via multicomponent condensation of aryl aldehydes, β-naphthol, and urea or acetamide. In_2O_3 nanopaticles were found to be highly active for the transformation with excellent yield and purity of the product in a short reaction time.

Keywords amidoalkyl naphthol, indium oxide, multicomponent condensation, nanocatalyst

INTRODUCTION

Over the past decade, research on nanocrystalline materials has been greatly centered on manipulation of structures on the molecular or atomic level. However, most of the studies have been directed to the synthesis, characterization, and applications as structural and optical/electronic materials. As catalysts, nanoclusters have been studied for a long time, but they are mainly limited to supported metal systems.[1] Direct synthesis and successful stabilization of nanocrystalline metallic and ceramic materials have only recently been investigated in detail for some catalytic applications.[2] Nanocrystalline metal oxides as a catalyst in different areas of the organic chemistry have now reached significant levels, not only for the possibility to perform environmentally benign synthesis, but for the good yield.[3–5]

Indium oxide (In_2O_3) has been synthesized by various physical and chemical methods and studied for many of its potential applications in solar cells,[6] PDP,[7] semiconductor gas sensors,[8] and photocatalysts,[9] Pulse laser abalation,[10] spray pyrolysis,[11] sol-gel method,[12] and hydrothermal synthesis[13] are a few to mention that hich were commonly used for synthesis of nanocrystalline In_2O_3. Recently Maensiri et al. reported the environmentally benign method of synthesis of In_2O_3 using indium acetylacetonate and aloe vera plant extract.[14] However, many of these methods require either special equipments or expensive chemicals or they are time consuming as well as tedious. The gel-combustion method[15] is now gaining lot of importance due to its advantages such as fast kinetics of the process (instantaneous) and high purity, homogeneity, crystallinity, tunable and fixed composition, and structure of products. Moreover, use of nontoxic precursors (if at all possible), relatively few numbers of reagents, and low temperature are plus points of the technique. In this technique, stoichiometric amount of metal nitrates (oxidizer) and fuel (reducing agent) are used. The combustion of the aqueous mixture results into generation of high exothermicity to obtain ultrafine particles of corresponding oxides.

Multicomponent reactions (MCRs) have attracted considerable attention because they are performed without isolating any intermediate during their processes; this reduces time and saves both energy and raw materials.[16] They have merits over two-component reactions in several aspects including the simplicity of a one-pot procedure, possible structural variations, and building up complex molecules.

Amidoalkyl naphthols can be prepared by MCR. It was reported that 1- amidoalkyl-2-naphthols was prepared by multicomponent condensation of aryl aldehydes, 2-naphthol, and acetonitrile or amide in the presence of Lewis or Brønsted acid catalysts such as montmorillonite K10 clay,[17] $HClO_4$-SiO_2,[18,19] iodine,[20,21] $K_5CoW_{12}O_{40}.3H_2O$,[22] *p*-TSA,[23] sulfamic acid,[24,25] and cation-exchange resins.[26] The main drawback of many of these reactions is that they require high amount of catalyst and due to this, they require large amount of organic solvents which are volatile and toxic in nature. Ionic liquids are one of the alternatives[27,28] to employ catalysts or catalytic reagents as selective as possible and are superior to stoichiometric reagents.

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Address correspondence to Vishvanath D. Patil, Department of Chemistry, C.K. Thakur College, Plot 3A, Sec. 10, Khanda Colony, New Panvel (w), Raigad, Maharashtra, India 410206. E-mail: patilvd148@yahoo.in

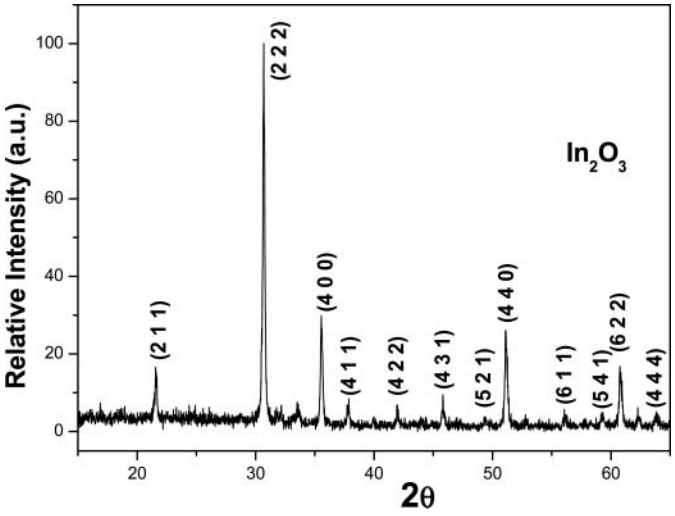


FIG. 1. XRD pattern of synthesized nanocrystalline In_2O_3.

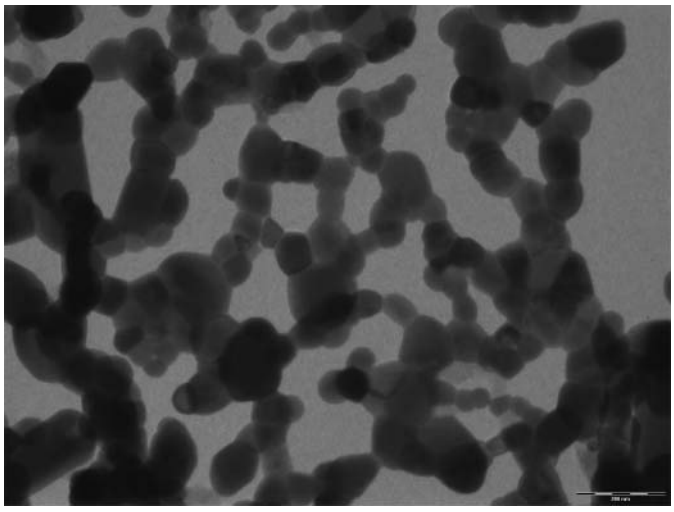


FIG. 2. TEM image of synthesized nanocrystalline In_2O_3.

This study demonstrates synthesis of nanocrystalline In_2O_3 by gel-combustion method, characterization by powder X-ray diffraction (XRD), and its scope as a catalyst in the novel and rapid synthesis of various amidoalkyl napthols as compared to the commercially available In_2O_3

EXPERIMENTAL

Synthesis of Nanocrystalline In_2O_3 Catalyst

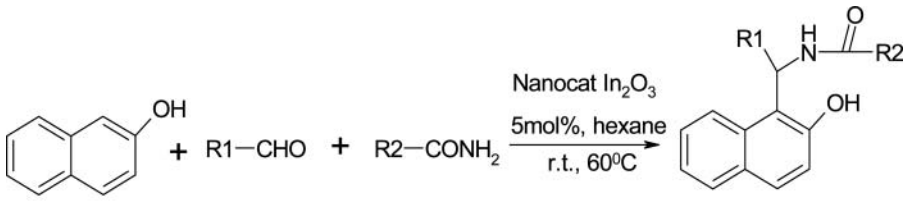
Gel-combustion synthesis[29–31] is a soft chemical route used to synthesize various nanocrystalline oxides. Stoichiometric proportion of indium nitrate (oxidant) and glycine (fuel), calculated on the basis of propellant chemistry[32] is used to synthesize indium oxide. Stoichiometric ratio of oxidant-to-fuel (1:1.66) is dissolved in a minimum amount of deionized water to obtain transparent aqueous solution. This solution is then heated at about ∼100°C on a hot plate to remove excess of solvent results in highly viscous gel. On further heating at ∼300°C, the gel gets swelled and undergoes combustion with flame and rapid evolution of gas to produce voluminous powder. The powder obtained was then calcined at 600°C for 1 h to remove carbonaceous impurities if any. The powder is then characterized by powder XRD to confirm the formation of single-phasic In_2O_3 and its crystallite size was determined by Scherrer's formula and morphology was studied by TEM.

Characterization of Nanocrystalline In_2O_3 Catalyst

X-ray diffraction measurements were carried for phase analysis and crystallite size estimation, using monochromatized Cu-Kα radiation on a Philips X-ray diffractometer, X'pert PRO (Almelo, The Netherlands). Silicon was used as an external standard for correction due to instrumental broadening. The crystallite size was calculated by Scherrer's formula.[33] The TEM micrographs were obtained with Phillips CM 200 transmission electron microscope (TEM; Eindhoven, The Netherlands). The preparation of samples for TEM analysis involved sonication in isopropanol for 5 min and deposition on a carbon coated copper grid. The accelerating voltage of the electron beam was 200 kV. The surface area analysis was carried out by the standard BET technique with N2 adsorption using a Quantachrome, Autosorb-1 analyzer (Florida, USA).

One Pot Synthesis of Amidoalkyl Naphthols Derivatives (General Procedure)

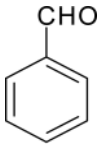
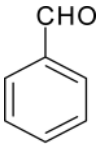
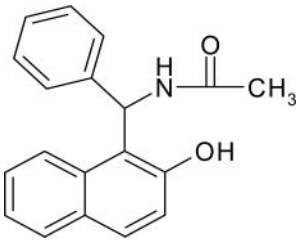
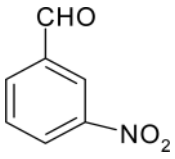
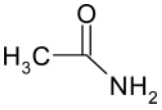
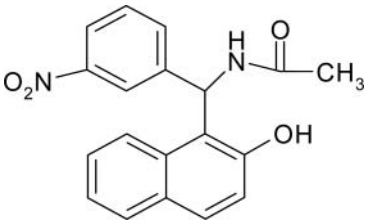
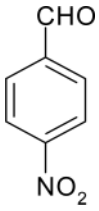
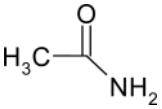
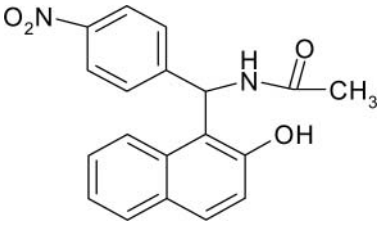
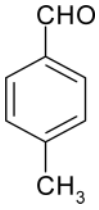
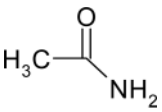
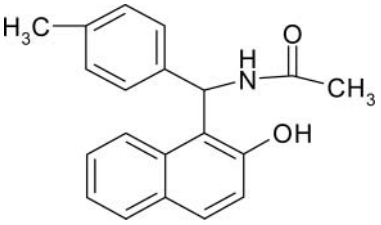
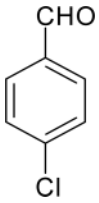
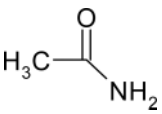
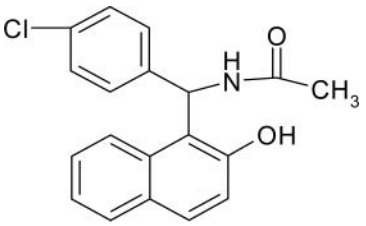
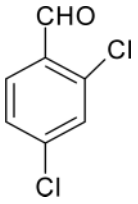
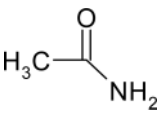
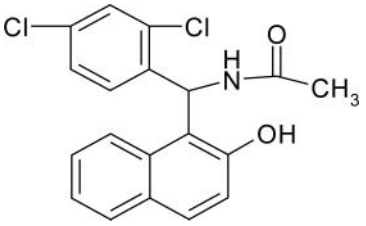
A mixture of 2-naphthol (2 mmol), m-nitrobenzaldehyde (2 mmol), acetamide or urea (2 mmol) and In_2O_3 bulk



R1= Alkyl, Aryl R2= Alkyl, -NH_2

SCH. 1. Synthesis of amidoalkyl naphthols.

TABLE 1
Synthesis of amidoalkyl naphthols by using catalyst as bulk and nanoparticles of In_2O_3

Entry	Aldehydes[a]	Acetamide/Urea	Product[b]	Bulk		Nanoparticles	
				Time (min)	Yield[c] (%)	Time (min)	Yield[d] (%)
1.				60	32	15	93
2.				70	35	10	95
3.				70	38	15	95
4.				90	43	15	89
5.				90	44	15	87
6.				120	33	15	85

(*Continued on next page*)

TABLE 1
Synthesis of amidoalkyl naphthols by using catalyst as bulk and nanoparticles of In_2O_3 (*Continued*)

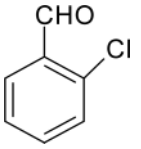
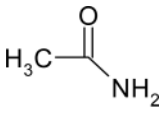
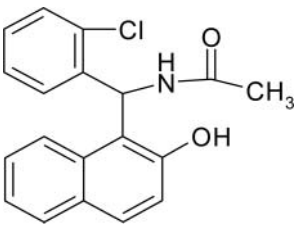
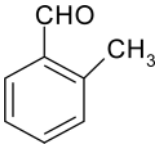
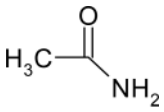
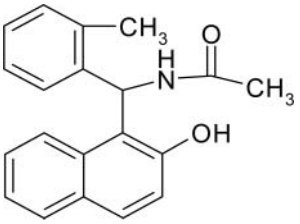
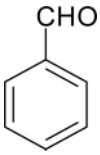
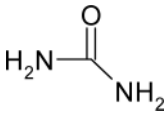
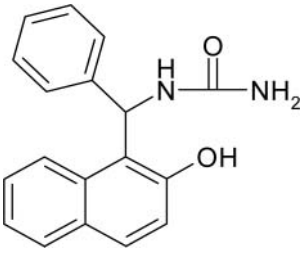
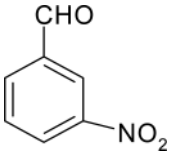
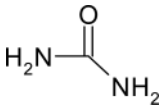
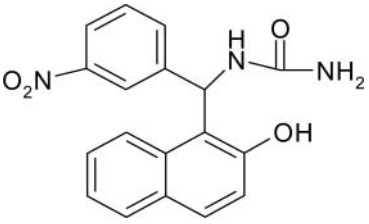
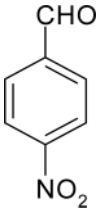
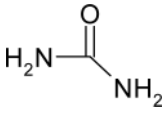
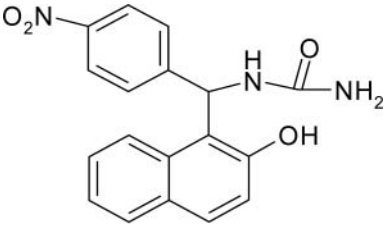
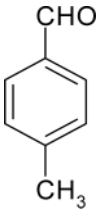
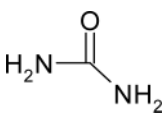
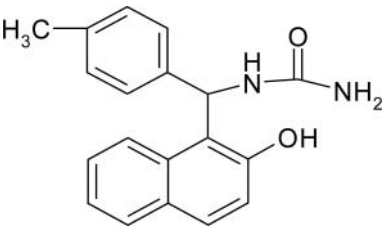
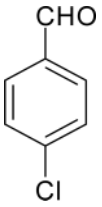
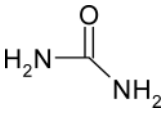
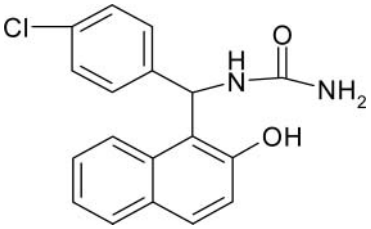
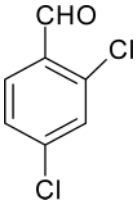
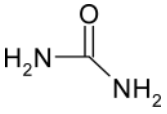
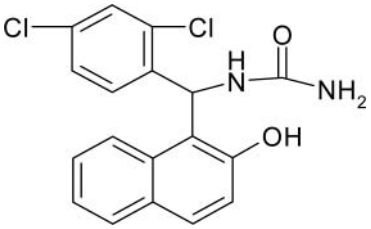
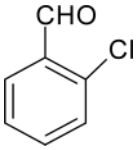
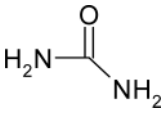
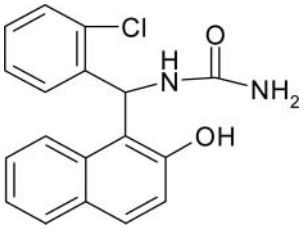
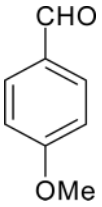
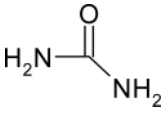
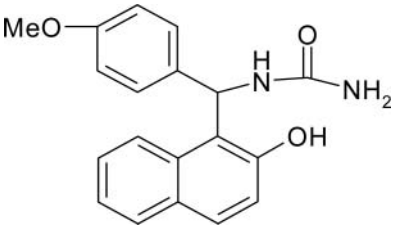
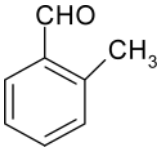
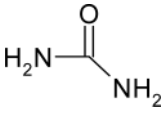
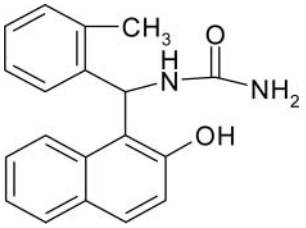
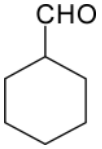
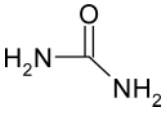
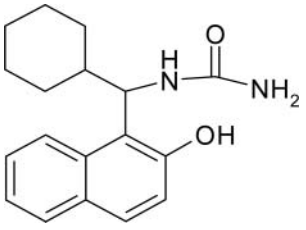
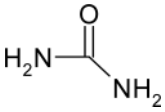
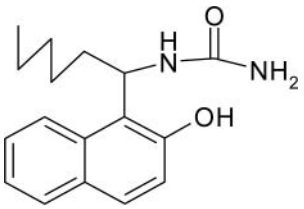
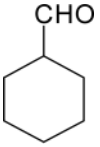
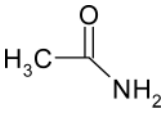
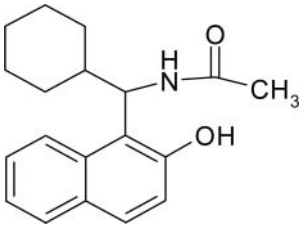
Entry	Aldehydes[a]	Acetamide/Urea	Product[b]	Bulk		Nanoparticles	
				Time (min)	Yield[c] (%)	Time (min)	Yield[d] (%)
7.				120	34	15	88
8.				90	46	15	90
9.				60	45	15	90
10.				70	48	10	96
11.				80	47	15	95
12.				90	41	15	91

TABLE 1
Synthesis of amidoalkyl naphthols by using catalyst as bulk and nanoparticles of In_2O_3 (*Continued*)

Entry	Aldehydes[a]	Acetamide/Urea	Product[b]	Bulk Time (min)	Bulk Yield[c] (%)	Nanoparticles Time (min)	Nanoparticles Yield[d] (%)
13.				90	43	15	90
14.				120	36	15	87
15.				120	38	15	90
16.				90	47	15	89
17.				90	50	15	92
18.				120	30	15	70

(*Continued on next page*)

TABLE 1
Synthesis of amidoalkyl naphthols by using catalyst as bulk and nanoparticles of In_2O_3 (*Continued*)

Entry	Aldehydes[a]	Acetamide/Urea	Product[b]	Bulk		Nanoparticles	
				Time (min)	Yield[c] (%)	Time (min)	Yield[d] (%)
19.				120	45	15	62
20.				120	25	15	65

[a]The substrate(2-naphthol) was treated with benzaldehyde (2 mmol) and actamide or urea by using 5 mol% of In_2O_3 nanoparicles and bulk in presence of hexane and at 60°C.
[b]All products were identified by their IR and NMR spectra.
[c]Isolated yields by using In_2O_3 bulk.
[d]Isolated yields by using In_2O_3 nanoparticles.

nanoparticles (5 mol%) was stirred magnetically at 60°C in the presence of hexane (1 mL) and the progress of the reaction was monitored by thin-layer chromatography (TLC). The reaction mixture was filtered and extracted with ethyl acetate (3 × 30 mL) and water (10 mL). The combined extracts were dried with Na_2SO_4 and concentrated under reduced pressure. In all the cases, the product obtained after the usual work up gave satisfactory spectral data.

For *N*-[(2-Hydroxy-naphthalen-1-yl)-(3-nitro-phenyl)-methyl]-acetamide (**2b**), pale yellow solid, IR (KBr): 712, 1348, 1412, 1522, 1655, 2973, 3086, 3395 cm^{-1}. ^1H NMR (DMSO-d6): δ (ppm) = 2.03 (s, 3H), 7.25–8.11 (m, 11H), 8.64 (d, J = 6.92 Hz, 1H), 10.15 (s, 1H). ^{13}C NMR (DMSO-d6): δ (ppm) = 23.6, 48.5, 118.5, 119.4, 121.1, 122.3, 123.7, 123.7, 127.7, 129.6, 129.6, 130.4, 130.5, 133.2, 133.5, 146.8, 148.6, 154.8, 170.2.

For *N*-[(2-Hydroxy-naphthalen-1-yl)-p-tolyl-methyl]-acetamide (**4b**), White solid, IR (KBr): 708, 1352, 1415, 1521, 1657, 2974, 3060, 3394 cm^{-1}, 1H NMR (DMSO-d6): δ (ppm) = 2.00 (s, 3H), 2.20 (s, 3H), 7.10–7.32 (m, 8H), 7.70–7.82 (m, 3H), 8.40 (d, J = 8.12 Hz, 1H), 9.95 (s, 1H). ^{13}C NMR (DMSO-d6): δ (ppm) = 21.2, 23.8, 48.6, 119.9, 120.1, 123.3, 126.5, 127.6, 129.4, 129.7, 130.9, 133.4, 135.7, 140.6, 153.7, 170.3.

RESULTS AND DISCUSSION

Nanocrystalline In_2O_3 was successfully synthesized by simple and cost effective gel-combustion method. The powder XRD pattern of as prepared In_2O_3 revealed that the product formed is single-phasic and all diffraction peaks are in agreement with the JCPDS card no. 06–0416 (Figure 1). The crystallite size was estimated from broadening diffraction peak by using Scherrer's equation,[19] which is found to be 48 nm. TEM image (Figure 2) showed chains of irregularly shaped In_2O_3 nanoparticles. The grain size is in agreement with the XRD results. The ultrafine grain size of the nanomaterials provides high surface-to-volume ratio. The morphological features show high population of atoms located at the edges, corners and on the surfaces of nanocrystallites provides active sites for catalyzing surface reactions.[34,35] Moreover, In(III) salts are known as Lewis acids. Their stability to co-ordinating atoms present in organic substrates makes them excellent catalysts.[36]

The specific surface area of the nanocrystalline In_2O_3 and bulk In_2O_3 was measured and it was found that nanocrystalline In_2O_3 has surface area 47.55 m^2/g whereas bulk In_2O_3 has surface area 1.02 m^2/g. High specific surface area of nanocrystalline In_2O_3 attributes to enhanced surface chemical reactivity than its bulk counterpart which in turn responsible for its high catalytic activity.

Here in this article we made an attempt to propose a novel protocol for the rapid synthesis of a variety of amidoalkyl naphthols using a catalytic amount of In_2O_3 Nanopaticles under extremely mild conditions (Scheme 1).

Optimization of Reaction Condition

To optimize the conditions, the reaction of benzaldehyde, β-naphthol, and acetamide was selected as a model to investigate the effects of different amounts of catalyst on the yield. To test the general scope and versatility of this procedure in the synthesis of a variety of substituted amidoalkyl naphthols, we examined a number of differently substituted aryl aldehydes, β- naphthols, and acetamide. The best result was obtained by carrying out the reaction with equal molar amounts of aldehyde, β-naphthol, and acetamide. The reaction mixture was stirred with 60°C in the presence of 5 mol% of catalyst.

All aliphatic and aromatic aldehydes bearing electron-donating or electron-withdrawing substituents gave corresponding amidoalkyl naphthols. However, aromatic aldehydes were more desirable in terms of yield. In case of aromatic aldehydes, it was observed that aldehydes with electron-withdrawing groups (Table 1, entries 2, 3, 9–11) gave higher yields than aldehydes with electron-donating groups (Table 1, entries 4–7, 12–17). Electronic and steric factors also decrease the yield of the product (Table 1, entries 6–8, 14, 15, 17).

Synthesis of amidoalkyl naphthols was selectively carried out by using In_2O_3 catalyst in the bulk form (commercially available) as well as nanocrystalline form. It was found that nanocrystalline In_2O_3 gave excellent yield in short period of time while bulk In_2O_3 gave relatively poor yield. The plausible reason behind this is the active surface, where the reaction takes place. The active surface increases as the size of the catalyst decreases, which ultimately results into the enhanced reaction efficiency. Thus, nanocrystalline In_2O_3 is highly efficient as a catalyst to obtain higher yield in the synthesis of amidoalkyl naphthols.

CONCLUSION

In conclusion, In_2O_3 nanoparticles synthesized by facile gel-combustion method were found to catalyze synthesis of amidoalkyl naphthols derivatives with high efficacy. The salient features of this method include a simple procedure, short reaction time, mild conditions, easy purification, generality, and in addition not cumbersome apparatus are needed. Nanostructured catalysts have a significant influence on the conversion and selectivity of the chemical reactions. Particle size and structure of the catalytic active species is the most important factor in understanding the difference between the catalytic properties of nanostructured catalysts and its bulk counterpart.

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