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Furfural condensation by using Zn-Mg-HT as solid base catalyst

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ABSTRACT
Hydrotalcite compounds like Zn-Mg-HT catalyst was prepared as by using the Co-precipitation route and characterized from a structural point of view, has helped the condensation of renewable resources.XRD analysis of synthesized catalyst represents the crystal quality of materials that helps to the reaction mechanism. The FESEM image shows the sheets like morphology of Zn-Mg-HT catalyst and the sheets of nanoparticles with average particle size of 40 nm. The reaction of renewable reactantto value added products as furfural: acetone mole ratio 1:10 was carried out, so as to form 4-(-2-furyl)-3-buten-2-on (FAc), 1, 4-pentadien-3-on-1,5 di-2-furanyl (F_2Ac) and 4-methylpent-3-en-2-on at 100°C.The reaction product(FAc), (F_2Ac), analyzed by using the GC-MS and confirms the mass of products.
Keywords: *FESEM, FAc, F_2Ac, Zn-Mg-HT etc*

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INTRODUCTION

Recently, the hydrotalcite synthesis has devoted to study of the reaction development in the field of more clean, efficient fine-chemical production methods. This helps to leading catalytic processes into one-pot synthesis. Now a day's researchers have tremendous efforts in the field of sustainable improvement of condensation reaction products, as fuels and chemicals from renewable biomass[1]. In growing field of biomass, the hydrotalcite catalyst has been widely used in the field of cross aldol condensation as solid base catalyst[1,2]. Hydrotalcite (HT) is the one of the best mixed metal oxide and has widely used in the field of condensation reaction due to their basic characters [3]. The condensation reaction of furfural with acetone is a well-known and it proceeds in the presence of hydrotalcite catalysts [4,5]. The solid base catalysts are used for reaction because it has high selectivity, activity and conversion efficiency of reactants.

The solid base crystalline hydrotalcite materials have strong approach towards the catalytic reactions. The large number of hydrotalcite materials developed as Mg-Al-HT [4], Zn-Mg-HT.Present work developed on the basis of solid base catalyst in normal synthetic route. The cheap and easy synthesis routes are developed so as to convert furfural into the value added products. The researchers have attracting focus on the synthesis of hydrotalcite materials as mixed metal oxides[2,7,8].

This paper emphasize on synthesis of advanced nanostructure, nanomaterials as hydrotalcite materials. The acceptable ability of catalyst with high conversion efficiency materials has a alternative route for large scale production of value added products in the green chemistry. The nanocrystalline catalyst synthesized by number of attempts to control, size, shape, crystal structure and composition of Zn-Mg-HT catalyst by co-precipitation method[9]. For high conversion of reactants into value added products, the reaction was studied with respect to reaction conditions. Further theZn-Mg-HT material was characterized by the FESEM, and XRD.

In this investigation, furfural: acetone (1:10) condensation product was (FAc), (F_2Ac) and 4-methylpent-3-en-2-on respectively. This reaction adapt environmental approach and hence a green reaction route. The Zn-Mg-HT catalyst can be reused and shows the reproducibility of results. The method of synthesis was simple and inexpensive.

MATERIAL AND METHODS

All the chemicals purchased from the reputed commercial brands like Avra synthesis, Sigma-Aldrich and S.D. Fine chemicals and used as without any further purification. The catalyst synthesis and reaction

methodology are explained stepwise manner. The analysis of reaction mass and catalyst characterization takes place by using characterization techniques.

The synthesis of Zn-Mg-HT (Zn/Mg = 2.5) as: The reaction mixture of $Zn(NO_3)_2 \cdot 6H_2O$ (0.0885 mol) (Aldrich and (0.221 mol) and $Mg(NO_3)_2 \cdot 6H_2O$ (0.2213 mol) (Aldrich) was added in NaOH (0.7162 mol) and Na_2CO_3 (0.2084 mol) in a round-bottom flask. The reaction time was 2 h and reaction mixture was heated to 338 K for 15 h. Then filter the solution so as to obtain precipitate. Then neutralize the precipitate by washing of hot distilled water and dried at 353 K in oven.

In present experiments, 1-2 g of Zn-Mg-HT catalyst was added with the reaction mixture of acetone and furfural (furfural /acetone 1/10) composition. The temperature of reaction (autoclave) was (T = 100⍰ C), and kept constant. The product was confirmed with GC-MS.

RESULTS AND DISCUSSION

XRD

The XRD pattern of hydrotalcite materials provides information about the crystalinity of the Zn-Mg-HT catalyst. Fig. 1

provides the XRD patterns of the Zn-Mg-HT. The broad sharp peak with low intensity was detected in the range of 20-65° in diffractograms of Zn-Mg-HT. These diffraction peaks at high 2θ denotes the presence of larger crystallites. The XRD data obtained for Zn-Mg-HT catalyst are in good agreement with good agreement with literature [5,10].

Each peak has been detected by Full Width Half Maximum (FWHM) for at specific 2θ degree.

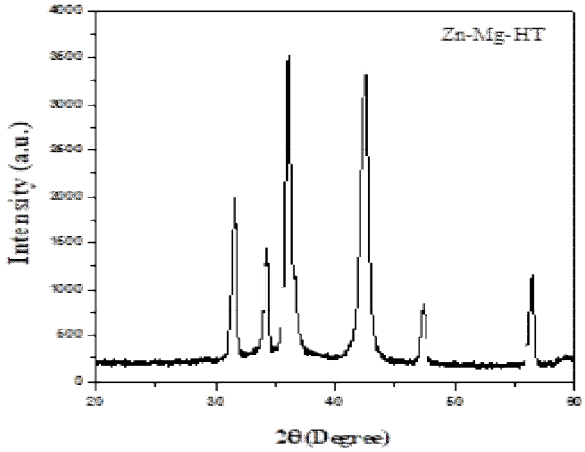

Figure 1:XRD patterns of the catalyst.

FESEM

The catalyst surface development and the nature of nanostructures with average size, morphology was scrutinized through FESEM. The Zn-Mg-HT image show the sheet like morphology as shown in Figure 2, and the sheets are composed of nanoparticles with average particle size of 40 nm. The 2 b shows the sheets connectivity and porus natures.

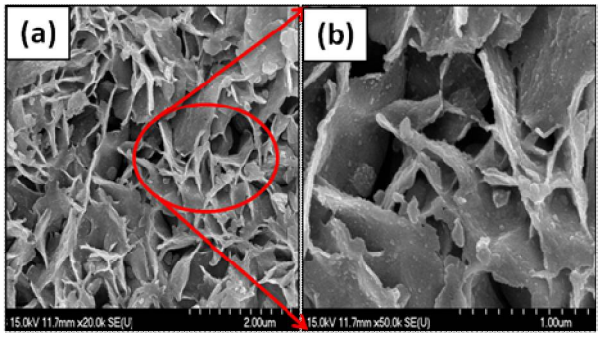

Figure 2: FESEM patterns of the catalyst.

GC-MS-Analysis

The condensation products of furfural/ acetone reaction confirmed by GC-MS as (FAc), (F_2Ac) and 4-methylpent-3-en-2-on etc. Figure 3 shows the GC-MS spectra of reaction. The reaction pathways and the products confirmations with the reported reaction are well matched.

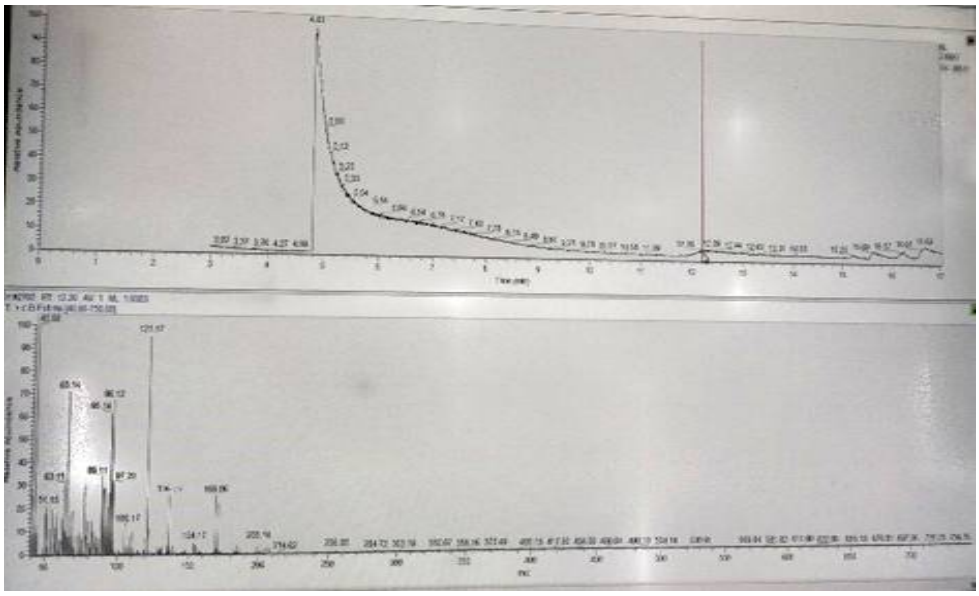

Figure 3: GC-MS Spectra of reported reaction.

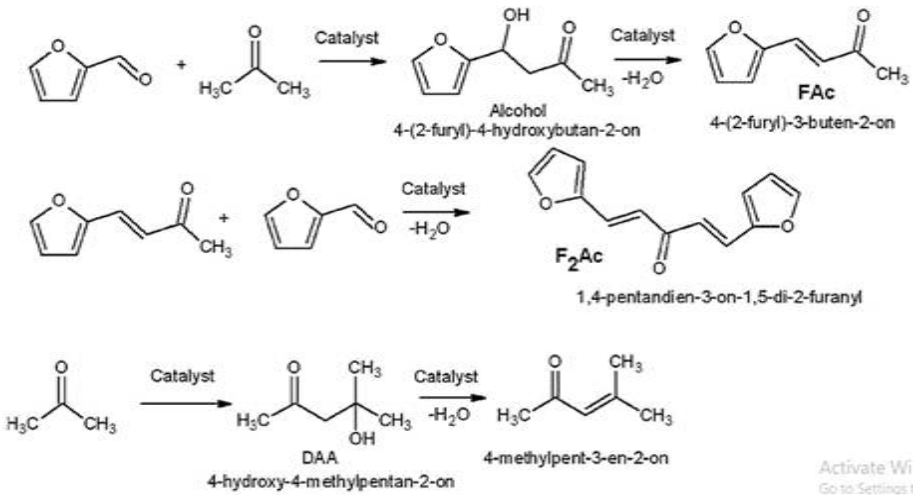

Fig 4: Furfural condensation.

CONCLUSION

In conclusion, Zn-Mg-HT catalyst synthesized by using the co-precipitation method and has best outcome in the field of catalysis. The furfural condensation reaction product was good agreements with the standard reaction products. The reaction pathways and the products confirmations with GC- MS data and the reported reaction are well matched.

The hydrotalcite materials have widely applicability in the field of green chemistry and value added chemical synthesis. Zn-Mg-HT has capacity to produce industrially valuable chemicals.

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