K-La-MgO as heterogeneous catalyst to synthesize of 3-(2-hydroxyethyl)-1, 3-oxazolidin-2-one from CO_2 and diethanol amine

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Abstract: 1,3-Oxazolidin-2-ones derivatives exhibits a wide application in pharmaceutical industries and various synthesis routes are employed to prepare these compounds which includes phosgenation, oxidative carbonylation, etc. making use of polluting chemicals and homogenous catalysts. The current work deals with developing a heterogeneous catalyst and its application to synthesize these derivatives using carbon dioxide as a C1 source. No use of promoter or organic co-catalyst makes the synthesis route more favorable. Pure La-MgO and K-La-MgO with different K loading were screened for carbonylation of diethanol amine and 5% K-La-MgO was found to be best catalyst.

Keywords: carbon dioxide, carbonylation, 1, 3-oxazolidin-2-ones derivatives

1. Introduction

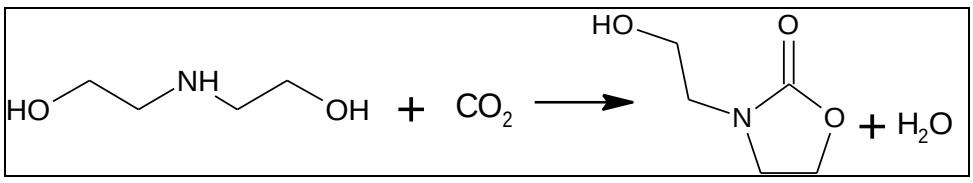
Fixation of carbon dioxide to industrially important chemicals has achieved greater attention due to environmental consideration as well as it is available at low cost in large quantities. Thus, developing specific catalysts to activate the carbon dioxide is a key for utilizing carbon dioxide chemically. Till date different carbonylating agents are also reported to synthesise these derivatives namely, isocyanates, urea, oxidative carbonylation using carbon monoxide and oxygen, dimethyl carbonate, etc. [1-3]. Replacing these carbonylating agents by carbon dioxide adds a green quotient to existing synthesis routes [4]. This excites to explore this synthesis route by developing a green heterogeneous catalyst to achieve carbonylation of diethanol amine without use of any noble metals and organic additives. We developed K-La-MgO (different loading of K) synthesized by combustion route and studied its application for carbonylation of diethanol amine with carbon dioxide.

In the current study, we focused mainly developing a heterogeneous catalyst and its application to synthesise urea derivative of diethanol amine using carbon dioxide as a feedstock. The catalyst was studied by different analytical techniques to have insight into textural and structural characteristics of catalyst.

2. Experimental

The catalysts namely, La-MgO and la-MgO with different K-loadings (1, 3, 5, and 7%) was prepared by combustion route and are well characterized by HR-TEM, XRD, TPD analysis and surface area analysis.

Reaction Scheme:



3. Results and discussion

HR-Transmission electron microscopy (TEM)

The HR-TEM analysis was done for virgin catalyst (5% K-La/MgO). The virgin catalyst (Figure 1a and 1b) showed irregular shape. The diffraction pattern of K-La-MgO catalyst shows concentric rings which indicate polycrystalline nature of the catalyst (Figure 1 c).

X-ray Diffraction (XRD)

The XRD pattern were recorded for La-MgO Figure 2 (a) and La-MgO with different K-loadings (wt%)1%, 3%, 5%, 7% and reused catalyst represented in Figure 2 (b), (c), (d), (e), and (f) respectively. In case of K- incorporated La-MgO (1, 3, 5, and 7%), the characteristic peaks for KNO_3 were absent confirms the absence of KNO_3 phase on the composite. While the characteristic peaks for K_2O (JCPD-03-065-2992) were observed suggests the dispersion of K as K_2O on the catalyst surface which provides the significant super basicity and high catalytic activity to the catalyst.

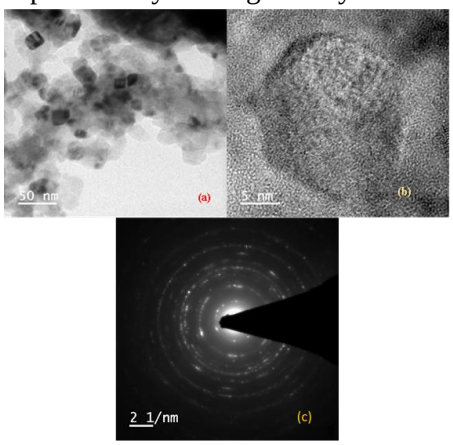


Figure 1. HR-TEM Images.

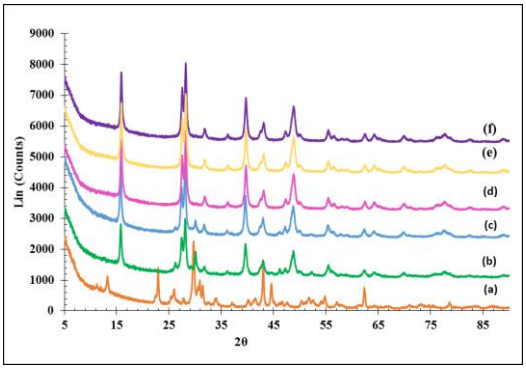


Figure 2. XRD Analysis

Efficacy of Catalyst was studied with different catalysts (Figure 3) i.e. K- incorporated La-MgO (1, 3, 5, and 7%), and 5% K-La-MgO was found best among screened catlysts. Effect of Carbon dioxide pressure was also studied (Figure 4) to intensify the conversion of diethanol amine.

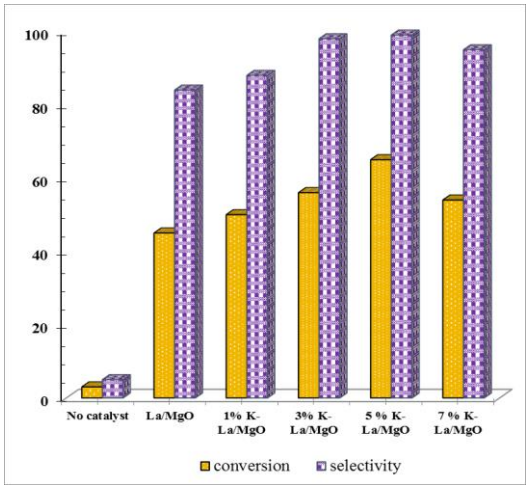


Figure 3. Efficacy of catalyst

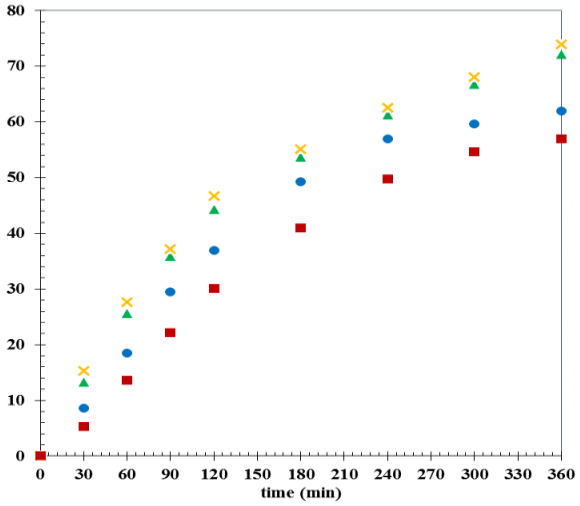


Figure 4. Effect of CO2 pressure on conversion of Diethanol amine

4. Conclusions

An alternative green route to synthesis 3-(2-hydroxyethyl)-1, 3-oxazolidin-2-one from diethanol amine and carbon dioxide without use of any organic promoter and co-catalyst was investigated. La-MgO and La-MgO with different potassium loading (1%, 3%, 5%, and 7%) were screened for above reaction. It was concluded that 5% K-La-MgO was best catalyst among the screened catalysts which gave 72% conversion of diethanol amine and 100% selectivity for 3-(2-hydroxyethyl)-1, 3-oxazolidin-2-one.

References

1. I. Chiarotto, M. Feroci, J. Org. Chem. 68 (2003) 7137–7139.
2. S. Fujita, H. Kanamaru, H. Senboku, M. Arai, Int. J. Mol. Sci. 7 (2006) 438–450.
3. R. Nomura, M. Yamamoto, H. Matsuda, Ind. Eng. Chem. Res. 26 (1987) 1056–1059
4. K. Tominaga, Y. Sasaki, Synlett. 2002 (2002) 307–309.