

## Reusable Gypsum Based Catalyst For Synthesis Of Glycerol Carbonate From Glycerol And Urea

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### ABSTRACT

In this study, the catalytic carbonylation of glycerol with urea in the presence of gypsum based heterogeneous catalyst is reported for the first time. Gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) is one of the two calcium sulphate minerals found in nature and also one of the waste materials produced from advanced material industrial processing plant. The effect of physical and chemical pre-treatment procedures on gypsum was investigated. To obtain the catalyst structure-activity relationship, the treated catalysts were characterized by means of several characterization techniques (*i.e.* XRD, TGA, BET surface area, SEM, FTIR,  $\text{CO}_2$ -TPD,  $\text{NH}_3$ -TPD and Hammett test). Tuneable physico-chemical properties of gypsum based catalysts were successfully prepared by varying the pre-treatment techniques, which later on contributed to the variation of catalytic activity toward glycerol carbonate formation from glycerol. The highest catalytic activity obtained was for catalyst consisting  $\beta$ - $\text{CaSO}_4$  phase where it produced 92.8% conversion of glycerol, 90.1% selectivity and 83.6% yield of glycerol carbonate, respectively. The gypsum catalyst is easily recoverable and reusable for subsequent cycles of reaction. Similar physico-chemical properties of fresh and used catalyst were confirmed through XRD, FTIR and Hammett test analysis. Besides, the mechanistic pathway of glycerol carbonate was confirmed through the formation of glycerol carbamate as intermediate compound which was further established through time online analysis study using  $^{13}\text{C}$  NMR and ATR-FTIR, respectively. The study also clearly supports conversion of waste into wealth while promising proper disposal of waste to produce value added product.

**KEYWORDS:** Glycerol; Glycerol carbonate; Gypsum; Heterogeneous catalyst; Pre-treatment

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