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Chemical Engineering Research and Design

journal homepage: www.elsevier.com/locate/cherd

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Process intensification and waste minimization in liquid–liquid–liquid phase transfer catalyzed selective synthesis of mandelic acid

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A B S T R A C T

Conversion of biphasic reactions into triphasic reactions can lead to process intensification, waste minimization and selectivity enhancement. Unlike liquid–liquid (L–L) PTC, the Liquid–Liquid–Liquid phase transfer catalysis (L–L–L PTC) offers high order of intensification of rates of reaction and catalyst reuse. The rate of reaction is remarkably enhanced by the catalyst-rich middle phase, which is the main reaction phase. Separation of catalyst can be done easily and the separated catalyst can be reused several times by using L–L–L PTC. This leads to waste minimization and other benefits of Green Chemistry. Mandelic acid and its derivatives are used for their dual activities as antibacterial and anti-aging agents. In this work, mandelic acid was produced by L–L–L PTC reaction of dichlorocarbene with benzaldehyde. Dichlorocarbene was generated in situ by the reaction of chloroform and sodium hydroxide in the presence of poly ethylene glycol (PEG) 4000 as the catalyst. The selectivity to mandelic acid was 98%. The reaction mechanism and kinetics model were established to validate the experimental data.

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Keywords: Multiphase reactions; Phase Transfer catalysis; Kinetics; Green chemistry; Mandelic acid

1. Introduction

Phase transfer catalysis (PTC) is a well established technique, which aims at cost and waste minimization (Yadav, 2005). PTC has proved to be a very important means of transcending phase barriers in heterogeneous systems. Although the use of agents for anion transfer has been mentioned in some early publications and patents (Jarrouse, 1951), the foundation of PTC were laid in the late 1960s and early 1970s by the studies of Makosza (1975), Starks (1971), Yadav (1975), and Brandstorm (1978). PTC has been applied to over 700 processes in a variety of industries such as intermediates, dyestuffs, agrochemicals, perfumes, flavours, pharmaceuticals and polymers (Naik and Doraiswamy, 1998; Starks et al., 1994; Sasson and Neumann, 1997; Yadav and Sharma, 1981; Yadav and Bisht, 2004, 2005). Application of PTC instead of traditional technologies for manufacture of organic chemicals provides substantial benefits for the environment (Makosza, 2000). One of the main benefits to adopt PTC reactions is that it requires mild operating condition and cheaper reagents. It is possible to carry out a variety

of hazardous reactions by using PTC in a safe and efficient way (Sharma, 2002).

PTC reactions have been studied as liquid–liquid (L–L) PTC, solid–liquid (S–L) PTC, gas–liquid (G–L) PTC, solid–liquid–liquid (S–L–L) PTC, and liquid–liquid–liquid (L–L–L) PTC including those with a synergistic combination of microwaves (Yadav, 2004; Yadav and Bisht, 2004, 2005). The main disadvantage of PTC in commercial applications is the need to separate the catalyst from the product in the organic phase. This can be eliminated by using L–L–L PTC in which high order of intensification of rates of reaction occurs. The middle catalyst-rich phase can be created between the organic and aqueous phase (Fig. 1), which contains the reactants through a proper balance of lipophilicity, hydrophilicity, interfacial tension, solubilities, phase equilibria, and density. This third liquid phase will be formed only when the catalyst has limited solubility in both organic and aqueous phase (Starks et al., 1994; Yadav and Naik, 2001). Most of the catalyst is found in the catalyst-rich middle phase, where the main reactions take place and the presence of this third phase leads to remarkable enhancement

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Received 16 September 2011; Received in revised form 21 December 2011; Accepted 13 January 2012

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doi:10.1016/j.cherd.2012.01.007