Ethyl lactate production by reactive distillation using bio-based lactic acid and ethanol

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The research project PERCAL addresses biorefinery processes for the organic fraction of municipal solid waste (OFMSW) with a strong focus on a diversification of the products. As part of the project, a process for the production of the eco-friendly solvent ethyl lactate was developed. Ethyl lactate was produced by reactive distillation from ethanol and lactic acid – two building blocks, which have received much attention in literature in the last decades and are available in industrial scale. Ethyl lactate production is especially interesting for processing low-cost lactic acid, as enantiomeric purity is not required. Lactic acid which was obtained from OFMSW (provided by PERCAL partner ATB) was used to produce ethyl lactate samples for the validation in final products (window cleaner and printing ink) by partner HAYAT.

Ethyl lactate is an environmentally benign solvent, which is readily bio-degradable. It exhibits very low toxicity and does not act ozone-depleting. Ethyl lactate shows favourable solvency properties for many applications. It has the potential to substitute petrol-based volatile organic compounds (VOCs) in a vast range of applications if production costs are reduced. (Pereira *et al*., 2011) Ethyl lactate is especially interesting for applications, where the release of solvent into the environment cannot be avoided such as household cleaning products, inks and paints. According to the EU emission inventory report, domestic solvent use and coating applications together accounted for 27% of the total non-methane volatile organic compound (NMVOC) emissions in the EU in 2017. (European Environment Agency, 2019)

Ethyl lactate was produced by the esterification of lactic acid with ethanol in a reactive distillation process. Reactive distillation combines the chemical reaction with simultaneous separation of the products by means of distillation in a single device. The mayor benefit of such a combined operation is the effect of process intensification. By constantly removing the side product water from the mixture, the equilibrium limitation of batch reactors is overcome and lactic acid conversion above the batch equilibrium is reached. The driving force for the chemical reaction is retained at a high level and the reaction rate is enhanced.

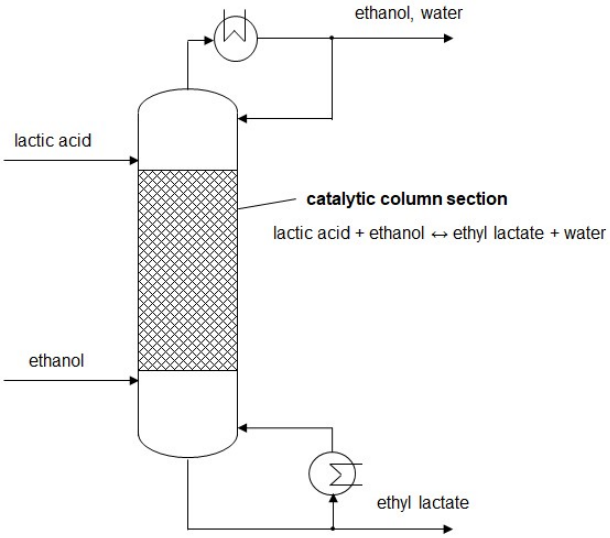

Figure 1. Schematic illustration of ethyl lactate production by reactive distillation

The production of ethyl lactate by reactive distillation has been previously studied by Asthana et al. (2005), who focused on the separation of ethanol and water from the bottom product. The goal of this work is the design of a scalable process with high product yield. An evaluation and optimization of the feedstock characteristics regarding the reaction kinetics was done in lab scale (0.1 kg ethyl lactate) to determine the process requirements and the reaction time required to reach sufficient lactic acid conversion. The mean residence time in the catalytic column section is essential information for the design of a scalable process. A reactive distillation prototype was used to scale-up the process to bench scale (3 kg ethyl lactate). The prototype has a column diameter of 50 mm

and a total height of 4.5 m. The central part is a catalytic column packing (Katapak-SP^{TM} 11, Sulzer) which contains the catalyst (Amberlyst 46, Dow). The initial water content of the feed mixture was reduced by concentrating a commercial lactic acid solution (88 w% solution, Carl Roth) to 96 w% in a rotary evaporator (R205, Büchi) at 2 kPa and 353 K before mixing with ethanol (99.9%, Australco) at a ratio of n_{EtOH}/n_{LA} = 3:1. Absolute ethanol (99.9%, Australco) was used as a stripping agent to remove water into the top product. The mean residence time in the catalytic zone was varied by recycling of the bottom product.

The scale-up of the reaction conditions was successful. The reaction kinetics in bench scale (3 kg ethyl lactate) was similar to the optimized lab scale operation (0.1 kg ethyl lactate). A lactic acid conversion of 89% was reached after 2.6 h reaction time. Product purity >90% ethyl lactate was reached when ethanol was distilled off in a rotary evaporator (R205, Büchi) at 10 kPa and 333 K. The obtained reaction kinetics was superior to similar processes in literature, where a lactic acid conversion about 90% was reached after 4 h. (Khunnonkwao *et al*., 2012; Tanaka *et al*., 2002) This can be explained by the reduced water content of the feedstock used in this work.

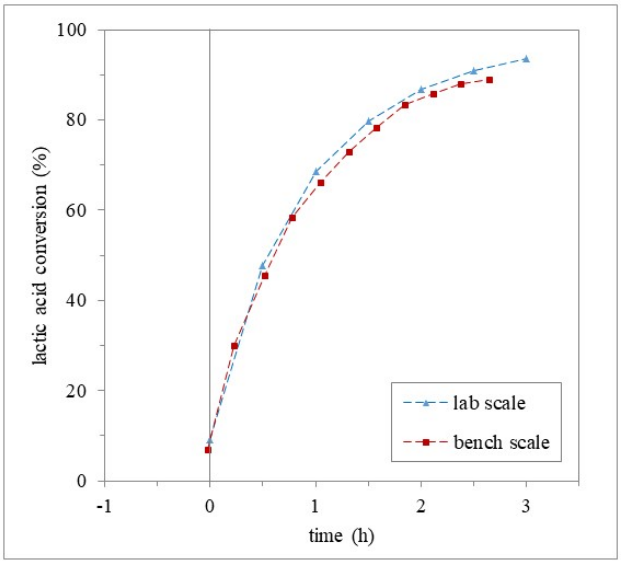

Figure 2: reaction kinetics of ethyl lactate production in lab scale (0.1 kg) and bench scale (3 kg)

An overall feed ratio of 3.81 kg ethanol/kg lactic acid was used. For an efficient and economic operation of the process a unit for the recovery of ethanol from the top product is required. Dehydration of ethanol is a well-established process in industry and economically viable in large production scale. It can be done either by adsorption of water on a molar sieve, or by using a membrane process such as pervaporation to separate water. Based on the results of PERCAL, process development will be continued. The next step will be to establish a continuous production process at pilot scale, before the integration into a biorefinery will be targeted.

Acknowledgements

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