SYNTHESIS OF POLYAMIDE-BASED MICROCAPSULES VIA INTERFACIAL POLYMERIZATION: EFFECT OF EMULSIFIER AND STIRRING RATE

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ABSTRACT

Polyamide microcapsules have gathered significant research interest during the past years due to their good barrier properties; however, the potential of their application is limited due to the fragility of the polymeric membrane [1,2].

Fully aliphatic polyamide microcapsules (PA MCs) were herein prepared from ethylene diamine (EDA) and sebacoyl chloride (SC) *via* interfacial polymerization in an oil-in water emulsion. The effect of the emulsifier type and content and stirring rate, were examined concerning attainable process yield and microcapsule properties (shell molecular weight and thermal properties, MC size and morphology). Two commercial emulsifiers with different molecular weight ranges and similar hydrolysis degrees were examined at 0.5 and 1 % wt. contents.

As the controlling step of the process is the diffusion of the amine component through the membrane in the organic phase, the partition coefficient of EDA in three organic solvents (dodecane, hexane and toluene) was experimentally measured. Toluene was selected as the most prominent solvent for MCs synthesis.

Turning on the effect of emulsifier molecular weight, the use of higher MW (130000 g mol^{-1}) yielded MCs with more complete shell structure, higher shell molecular weight ($0.23 \leq [\eta] \leq 0.61$ dL g^{-1}) and higher process yield (≥68%). This is probably due to a decrease in the interfacial tension between the organic and the aqueous phase, therefore increasing emulsion stability. In the case of the lower MW range (30000-70000 g mol^{-1}) the increase of the emulsifier concentration from 0.5 to 1% did not affect the average MC size (38 μm) but led to a decrease in the polydispersity index (PDI).The same, was also noted for high MW emulsifier, but in this case the average MC size was larger (53 μm). This behavior has been previously reported in literature and is correlated to the decrease of the coalescence frequency of organic droplets [4]. As to the effect of stirring speed during the polymerization step, its increase from 100 to 400 rpm led to an increase in shell molecular weight as amine diffusion was aided, but a decrease in MCs size was noted and is apparently due to the more frequent breaking of evolving MCs.

KEYWORDS: polyamide microcapsules; interfacial polymerization; encapsulation; microcapsule morphology

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