

# Polyurethane Microcapsule with Different Structure Containing Fragrant Materials

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## 1. Introduction

The polyurethanes are an interesting polymer group with widespread characteristics by molecular composition[1,2]. Especially, polyether polyurethanes have been greatly studied due to their excellent physical superior blood compatibility[3,4]. Increase of the hard segment content in polyurethane structures could give polymers with higher elastic modulus, and polyurethanes with modified properties could be properties could be produced by molecular weight and chemical structure of soft segments and the structure differences of chain extenders [5].

The aim of this study is to evaluate the effects of the ratio of hard and soft segments in the polyurethane membrane as a wall-forming material, by changing the molecular weight of polyol, and the effects on particle size distribution and morphologies of the resulting microcapsules, with molecular weight similar to the core materials, through the polymer wall indirectly.

## 2. Experimental

### 2.1 Preparation of microcapsules

#### 2.1.1 Materials

Segmented polyurethane microcapsules were prepared with 2,4-toluene diisocyanate (TDI) as aromatic polyisocyanate, poly(ethylene glycol)s (PEG, Mw 400, 600, 1000, 2000) with high molecular weight as polyols, and ethylene diamine(EDA) as chain extender. Poly(vinyl alcohol) as a protective colloid and 1,4-diamine anthra quinone (DAA) and migrin oil as a core material were used without any further purification.

## 2.2 Preparation

Polyurethane microcapsules were synthesized by interfacial polymerization in an aqueous poly(ethylene glycol) dispersion with ethylene diamine as the chain extender of toluene diisocyanate in perfume oil using poly(vinyl alcohol) as the stabilizing agent.

## 2.3 Characterization

Infrared spectra of microcapsules were obtained with Nicolet Impact 400D FT-IR.

Mena particle size and distribution of the microcapsules were evaluated by a light-scanning technique.

Scanning electron microscopy was performed using a JSM-5400.

## 3. Results and discussion

### 3.1 structure of micorcapsules

FT-IR spectra of migrin oil and synthesized microcapsules containing migrin oil are presented in Fig. 1. As seen in the Fig, the spectrum shows absorption bands at  $1740\text{--}1700\text{cm}^{-1}$  for the C=O stretching of urethane and at  $1690\text{--}1650\text{cm}^{-1}$  for urethane-urea formation. The N-H stretching was observed at  $3450\text{--}3300\text{cm}^{-1}$ . The IR spectrum also indicates the completion of the reaction between diisocyanate and polyol by the disappearance of the NCO absorption band at  $2270\text{cm}^{-1}$  and appearance of the N-H and C=O absorption bands. Moreover, C-C stretching in the phenylene ring at  $1600\text{cm}^{-1}$ , =C-H stretching at  $2850\text{cm}^{-1}$ , C-O-C stretching at  $1100\text{cm}^{-1}$ , and =C-H bending at  $880\text{--}750\text{cm}^{-1}$  are also observed.

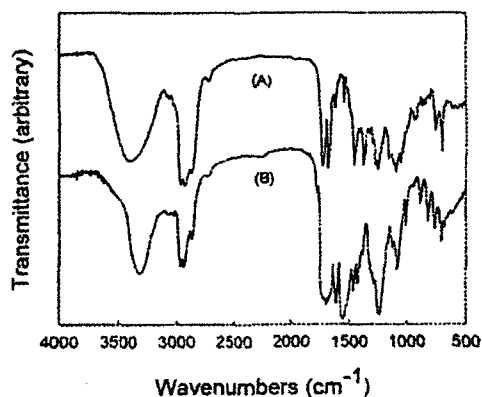


Fig. 1. FT-IR spectra of (A) migrin oil and (B) polyurethane microcapsules.

### 3.2 Particle size distribution

Fig. 2 shows the particle size distribution of polyurethane microcapsule with different molecular weight of PEG (400, 600, 1000, 2000). The mean size of the prepared particles increased up to 7.6, 8.2, 9.7, and 12.4 $\mu\text{m}$ , and the size distribution became wider with molecular weight. In the process of microencapsulation, globules in the first emulsion step would be the same size, but agglomeration could occur between the globules by adding polyol with diverse molecular weight crease of viscosity in the o/w emulsion. Thus, microcapsules from PEG-1000 and PEG-2000 have bimodal size distribution for this reason.

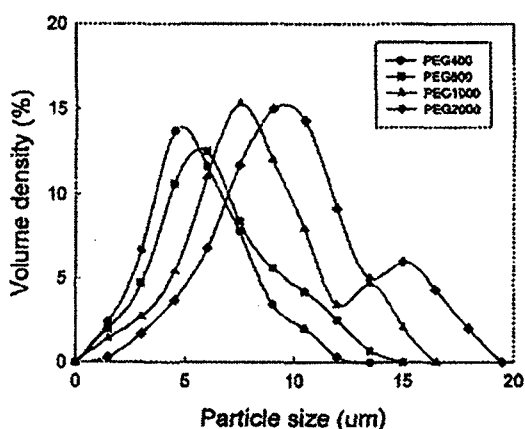


Fig. 2. Particle size distribution of high molecular weight polyol-based polyurethae microcapsules.

### 3.3 Morphologies of microcapsules

Fig. 3 shows morphologies of polyurethane microcapsules with different molecular weights of PEG. As a result, the more the molecular weight of polyether polyol increase, the smoother the surface of its microcapsules seem. The surface of microcapsules from PEG-400 seem so coarse and porous, while that from PEG-2000 seem smooth and less permeable. Eventually, the surface of the prepared microcapsules became more and more smooth with the molecular weight of PEG. It seems that a wider and thicker microcapsule wall was formed by formation of the soft segment with a longer chain by means of reaction between TDI and higher molecular weight polyol.

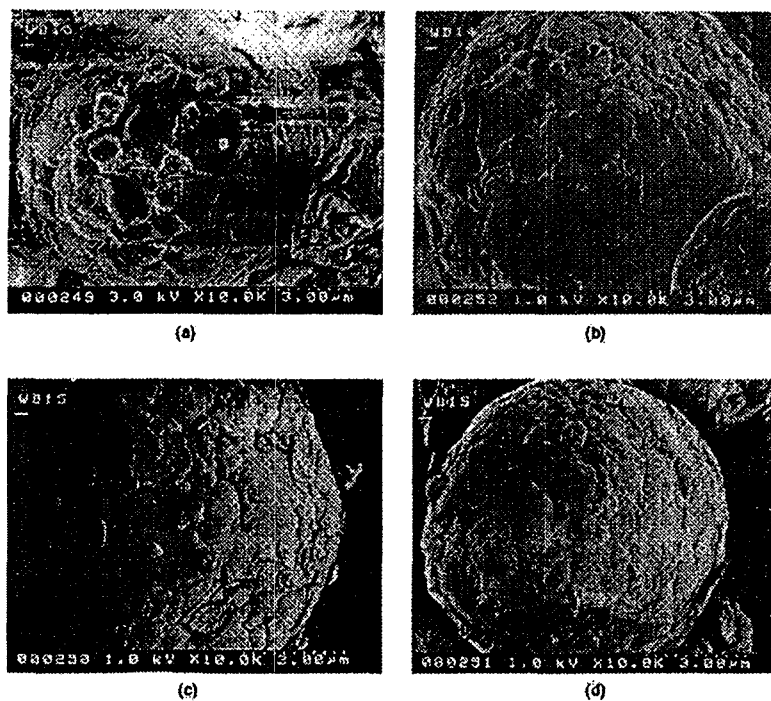


Fig. 3. SEM photographs of high molecular weight polyol-based polyurethane microcapsules : (a) PEG-400 (b) PEG-600 (c) PEG-1000 (d) PEG-2000

### 3.4 Release behavior

The release profile of disperse dye from microcapsules with different molecular weight of polyol in methanol are presented in Fig. 4. The initial dye concentration from microcapsules increases in the order of PEG-2000 > PEG-1000 > PEG-600 > PEG-400. The amount of encapsulated dyes seems to have to do with an increase of viscosity in the already formed o/w emulsion with addition of polyol on the preparation process. The release rate of DAA through microcapsules from PEG-400 is the fastest, even the smallest burst released content through the walls, whereas those from PEG-2000 are the opposite to PEG-400. Microcapsules with PEG-2000 seem non-permeable in that concentration of the released DAA hardly changes even with dissolution time, which results from a decrease of permeability of the hydrophilic soft segment in polymer membranes and their thickness. Eventually, sustained release properties could be controlled by various hard / soft segment ratios.

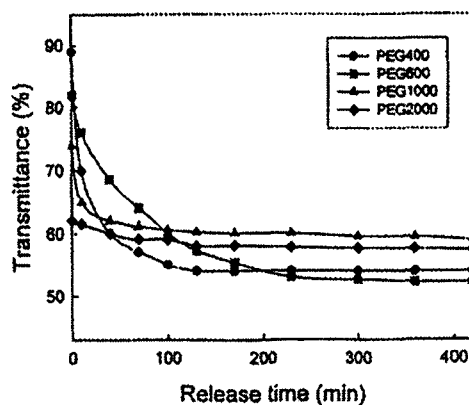


Fig. 4. Release behavior of DAA through molecular weight polyol-based polyurethane microcapsules in methanol.

### 3. Conclusion

Polyurethane microcapsules were synthesized by emulsion polymerization using TDI, polyether polyols and EDA, and characterized for their particle size distribution, morphologies, and release behavior of penetrator when produced with polyols of different chemical structures. A decrease in the molecular weight of polyol could give sharper, more porous and permeable micro-membranes due to an increase of the hydrophobic hard segment and their thinner wall. Sustained release behavior of microcapsules could be controlled successfully by the ratio hard / soft segments.

### References

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