

microcapsules containing migrin oil and 1,4-DAA. The resultant microcapsule slurry was decanted and washed with 30% ethanol to remove migrin oil and 1,4-DAA and unreacted isocyanates on the surface, and dried in vacuum oven at 25°C for 24 hours.

### 2.3. Characterization

Particle size distribution was taken using Galai CIS-100 (Galai Production Ltd., Israel). Scanning electron microscopy was performed using a JSM-5400 (JEOL Co. Ltd., Japan). Loading content of the penetrator, DAA from 0.6 g polyisocyanates microcapsules with different molar ratio of diisocyanates were obtained by adding into 500 ml of *N,N'*-dimethyl acetamide(DMAc) with stirring and kept at 20°C. The solution was assayed for the amount of the released 1,4-DAA by utilizing UV/visible spectrophotometer.

### 2.4. Preparation of fragrant fabric

Washing durability of fragrant fabric was investigated up to 15 times, after their preparation by printing of fragrant microcapsules, acrylic binder and water. The surface and durability of fabric was observed by SEM.

## 3. Results and discussion

### 3.1. Morphologies

Fig. 1 shows SEM photographs ( $\times 15,000$ ) of polyisocyanates microcapsules with different molar ratio of IPDI and TDI. Microcapsules surface from only TDI became rougher than any other samples and their wall seems thicker. This is mainly due to more active reaction on emulsion globules by addition of aromatic TDI with superior reactivity. Surface of microcapsule with same molar ratio of IPDI and TDI is much smoother than one with only TDI. It is considered that addition of aliphatic IPDI with inferior reactivity as wall material determines roughness of the microcapsules.

### 3.2. Particle size analysis

Figure 2 presents particle size distribution of PI microcapsules with different molar ratio of diisocyanates. Mean size of the microcapsules with increase of TDI content increased to 3.3 $\mu\text{m}$ , 3.8 $\mu\text{m}$ , 4.4 $\mu\text{m}$ , and 5.6 $\mu\text{m}$ , respectively and size distribution became broader. It is likely that this is related to reactivity of TDI which gives urea linkages with high probability forming

hydrogen bond, regardless of decrease of viscosity by lower molecular weight of TDI, compared with IPDI. Therefore, microcapsule wall from TDI by reactivity superior to aliphatic IPDI became thicker, although the size of emulsion globules seemed both same.

### 3.3. Loading content

Table 1 shows transmittance of penetrator, DAA in polyisocyanates microcapsules after dissolution in DMAc to know relative loading extent of penetrator and migrin oil. The DAA content in the resulting microcapsules became high more and more as TDI content with a lower molecular weight is higher than IPDI content with a higher molecular weight regardless of its larger globule size. This shows that TDI enhances reaction on globule surface due to its reactivity superior to IPDI, thus retention of penetrator and core material in microcapsules with higher TDI content is higher. This seems related to loss of core material and penetrator during microencapsulation process at strong stirring by a slow polymerization from IPDI.

### 3.4. Washing durability of fragrant fabric

Fig. 3 presents SEM photographs of the fragrant fabrics after 0, 10, and 15 times of washing test. As seen in the figures, the washing durability of the fabrics by printing is convinced of great for the industrial applications.

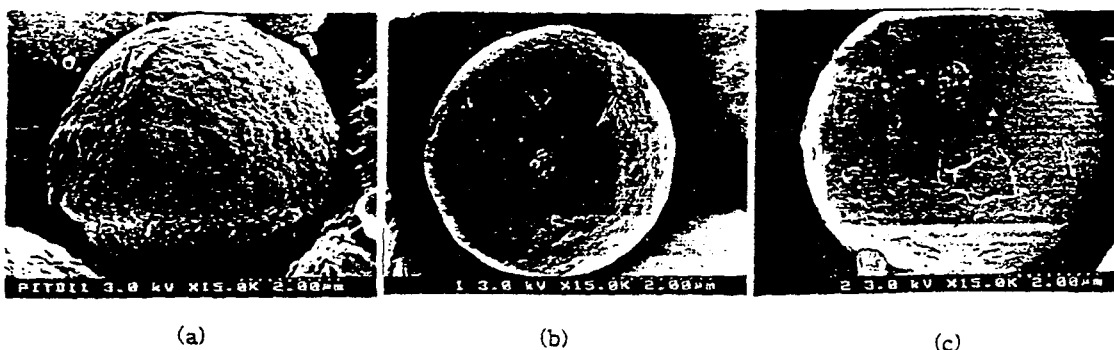


Fig. 1. SEM photographs of polyisocyanate microcapsules with different molar ratio ( $\times 15,000$ ): (a) only TDI, (b) same TDI:IPDI, and (c) only IPDI.

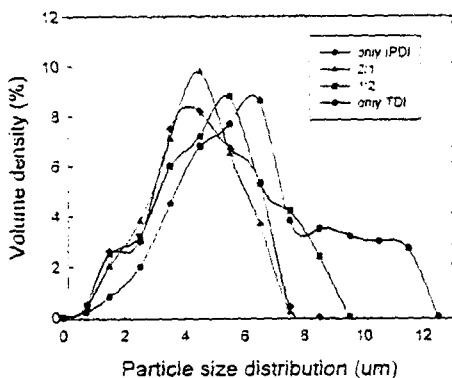


Table 1. Transmittance of 1,4-DAA in polyisocyanate microcapsules with different mixing molecular ratio

molecular ratio		Transmittance, %
IPDI	TDI	
IPDI only		85
2:1		51
1:1		50
1:2		41
TDI only		41

Fig. 2. Particle size distribution of polyisocyanate microcapsules with different molar ratios of IPDI and TDI.

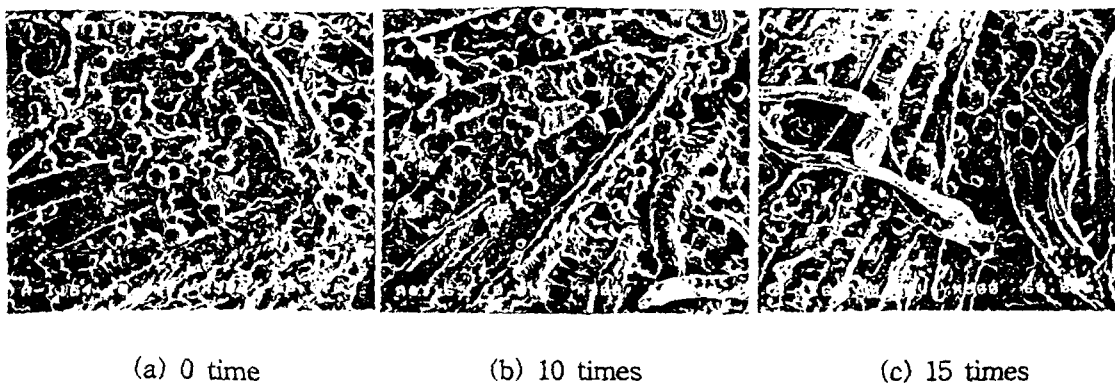


Fig. 3. SEM photographs after laundry test of fragrant fabrics printed with microcapsules

#### 4. Conclusion

Microcapsule wall from TDI by reactivity superior to aliphatic IPDI became thicker, regardless of decrease of viscosity by lower molecular weight of TDI, compared with IPDI. Penetrator loading content decreased with content of aliphatic IPDI in wall due to its reactivity inferior to aromatic TDI. Washing durability of the fragrant fabrics by printing was good for the industrial applications.

#### 5. References

1. H. Kataoka et al., JAPAN PATENT 09012447 (1997).
2. R. Gras et al., EUROPE PATENT 787754 (1997)