

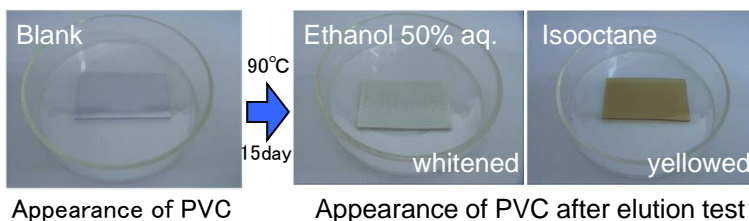
# Eluted materials from PVC sheet during chemical-resistant test, and its degradation.

X-6

Desorption of additives and degradation of polymer occur when plastic products contact to various liquids, such as a water and organic solvents. Consequently some components are eluted in the liquid. LC/MS/MS and spectroscopic techniques are powerful tools for qualitative and quantitative analysis of the eluted materials, and they could bring information on mechanisms of yellow discoloration.

## Analysis of elution of additives by LC/MS/MS

Polymer : PVC (1mm thick., 50mm × 30mm)  
Additives : DEHP (Di(2-ethylhexyl) phthalate) 2 %,  
TinuvinP (UV absorber) 0.015 %  
Solvent : Isooctane, Water, Ethanol 50% aq.,  
(70mL) Acetic acid 5% aq.  
Temp. : RT, 60°C, 90°C



Appearance of PVC

Appearance of PVC after elution test

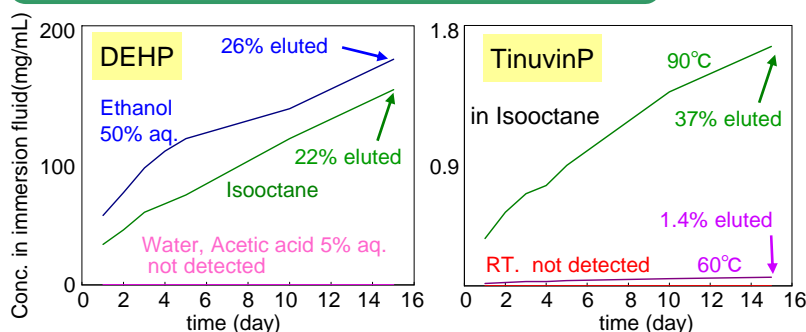


Fig. DEHP eluted at 90°C (left) and TinuvinP eluted by isooctane (right)

### Amount of eluted DEHP

Acetic acid  $\approx$  Water  $\ll$  Isooctane  $<$  Ethanol 50% aq.

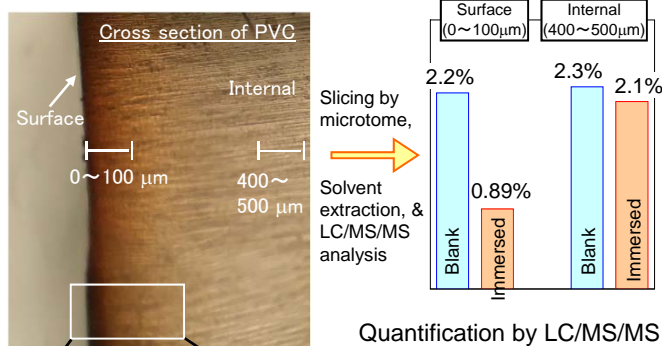
### Temp. dependency of elution of TinuvinP

The eluted TinuvinP increases rapidly over 60°C. It seems that the glass trans. temp. is important.

The quantity of eluted materials depends on the kind of the immersion fluid and temperature. The elution behavior can be analyzed by LC/MS/MS.

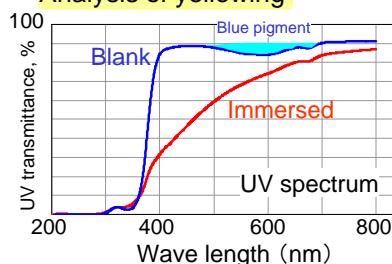
## Composition, structure, and properties of PVC immersed in isooctane at 90°C for 15 days

### Conc. of DEHP in depth direction



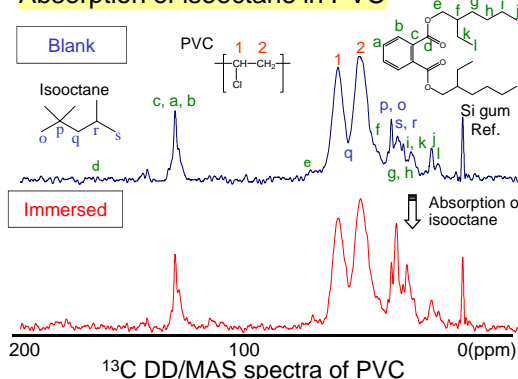
Quantification by LC/MS/MS

### Analysis of yellowing



It was estimated that the yellowing is caused by generation of the conjugate double bond in PVC by dechlorination and oxidation.

### Absorption of isooctane in PVC

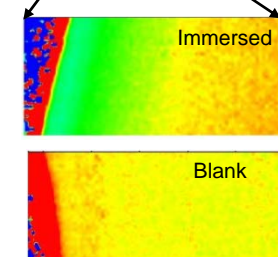


### Durometer Hardness

High  $\rightarrow$  softening  $\rightarrow$  Low  
Blank Immersed

### Glass trans. temp.

69.0°C plasticization 63.7°C  
DSC 1st. heat. 69.9°C desolvation 75.9°C  
Blank Immersed



Abs. Intensity  
1730cm<sup>-1</sup>/2910cm<sup>-1</sup>

### IR absorption imaging of ester bond C=O

※ including any ester bonds but DEHP

Quantitative analysis of additives in thin layer can be carried out by using microtome slicing and LC/MS/MS. DEHP amount in the internal of PVC did not decrease after immersing, whereas DEHP in the surface layer decreased by half, where yellowing occurred significantly.

The hardness of the immersed PVC decreased by processing plasticization caused by isooctane absorption.

Integrated analyses are necessary to comprehend degradation mechanisms of polymer. Degradation behavior of the polymer immersed in the solvent can be clarified by acquiring depth profile and bulk analyses with various techniques.