

Advanced Analysis of Yellowing Transparent Film

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1 Abstract

The market for transparent films for electronic devices and touch panels is expanding every year. To maintain transparency of the film over a long period, inhibition of yellowing is one of the most important problems^{1), 2)}. In order to develop an advanced transparent film, the yellowing mechanism has to be elucidated. However, the analysis of yellowing compounds in the film is difficult owing to the small amount. So, we developed a new analytical method to clarify the structure of yellow compounds in this study. Our method uses one of two different techniques based on whether the yellow compounds can be extracted by a solvent or not. In both cases we succeeded to clarify the structure of yellow compounds using the established methods.

2 Feature of analysis technique

- Analysis technique for yellowing compounds that is adapted to analysis sample
- Analysis technique that can distinguish yellowing compounds from other constituents with high probability
- Method of structural analysis capable of evaluating minute amounts (100 ppm) of yellowing compounds

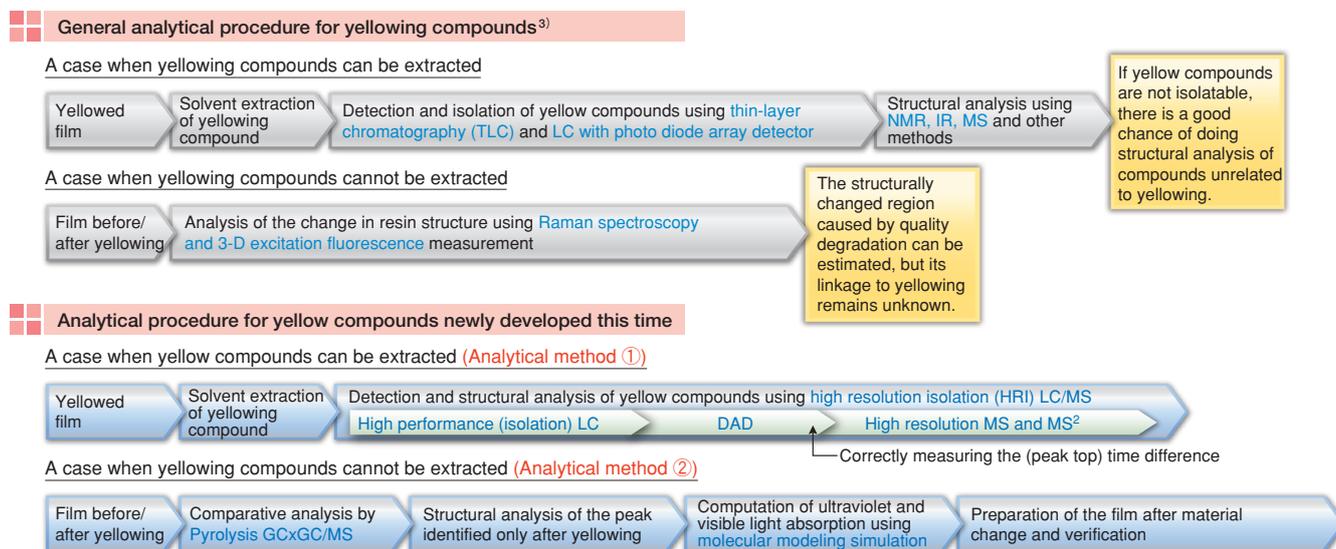


Figure 1 Comparison of a general analytical method of yellow compounds in yellowed film and the developed method

3 Background of the developments

Yellowed film that was the analytical target of this study was transparent film for use in touch panel and vehicle mounted display modules. Enough additives could not be used because transparent films for touch panel turned yellow after exposure. We received requests to elucidate the yellowing mechanism to help promote the development of films that can contain sufficient amounts of additive. In the case of transparent film for use in a vehicle-mounted display module, the mechanism of yellowing must be clarified in advance to make a policy decision on composition modification. So, we started development of analytical techniques applicable to each film to clarify the structure of yellowing compounds.

4 Technical details

In this study, we developed new multiple-track analytical procedures to meet situations when yellowing compounds can be extracted or not extracted as shown in **Figure 1**. Examples, where our developed analytical methods are applied, are shown below.

1) As an example for the case when yellow compounds can be extracted, we introduce an example of analytical procedures for yellowing compounds in transparent film for use in touch panels. When extracted solution of yellowing compounds was analyzed using an analytical method (1), an absorption peak at 440 nm was identified by photo-diode array detector (**Figure 2**). This

peak was considered as yellowing compounds and the MS spectrum to analyze was that located at a position with the same retention time shift between photo-diode array detector and mass spectroscopy (0.02 min. in this case). However, as shown from appearance of the mass spectral peaks, there were many compounds that eluted at the same retention time while each compound was separated by LC, so it was difficult to identify yellowing compounds from a particular m/z value. To distinguish between these m/z values, we wrote selected ion chromatograms for each m/z and determined the m/z with time shift of 0.02 min. between the selected ion chromatogram and the peak top at 440 nm as yellowing compounds. Once the m/z value to analyze was identified, we analyzed the MS² spectrum of that peak and determined the structure of a yellowing compound from fragmentation peaks. By correctly identifying the structure of yellowing compounds as exemplified in this case, we could figure out the structure of additives and blending ratios that would not produce yellowing compounds.

- 2) As an example of the case when yellowing compounds cannot be extracted, we introduce an example of analytical procedures used to identify yellow compounds in transparent film for use in a vehicle mounted display module. Only peaks specific to yellowing compounds in yellowed film were extracted after films before/after yellowing was analyzed by pyrolysis GC/MS (Figure 3). Then, we identified the structure of pyrolyzed material with help of the structural analysis of peaks appearing only in yellowed film, employing structural information from mass spectrometry. The determined structure of yellowing compounds can be considered as the one evolved during the weathering test. To deduce the intramolecular structure causing yellowing, we computed the absorption wavelength and oscillator strength of each compound by molecular modeling simulation (Figure 3). As a result, we found pyrolyzed material having the structure shown in Figure 3 could possibly be seen as yellow and deduced the structure of pre-weathering exposure chemical compound. To verify such a finding and estimation, we prepared a film not containing materials deduced to be the source of yellowing, and confirmed the lower yellowing level after weathering test, and no yellow compounds were detected by pyrolysis GCxGC-MS. Thus, even when yellowing compounds could not be extracted, we were able to estimate the source material of yellowing once a particular molecular structure of pyrolyzed material was determined as a source of yellowing and were therefore able to deduce the structure and blending ratio of additives that would not produce such yellowing compounds.

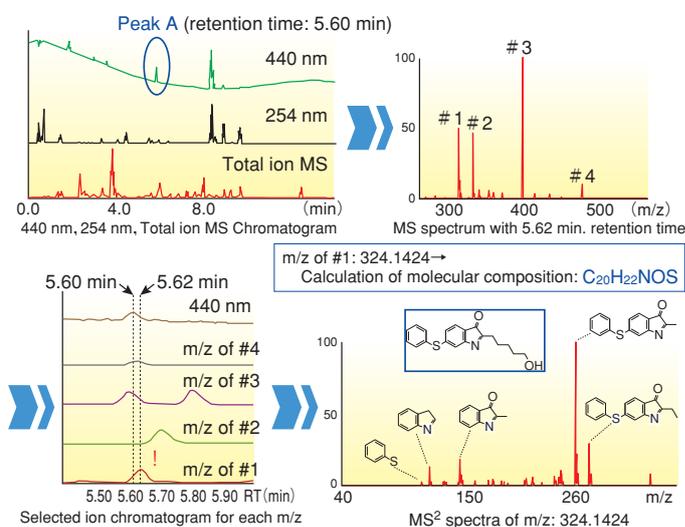


Figure 2 An analysis example of yellow compounds in yellowing transparent film for touch panels

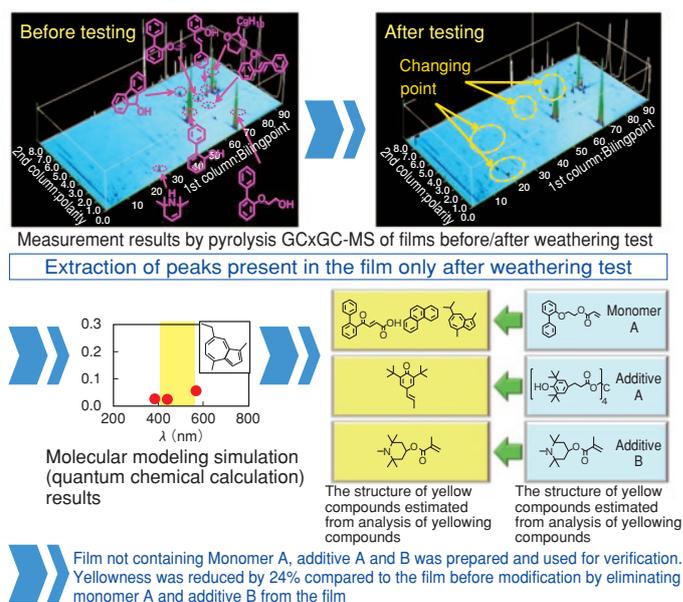


Figure 3 An analysis example of yellow compounds in yellowing transparent film for in-vehicle display modules

5 Future Business Development

- Development and applications of yellow compound analysis technique introduced herein to various transparent film
- Establishing an analytical technique to clarify the degradation mechanism by combining the methods introduced herein and multivariate analysis

[References]

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