Characterization of chitin-based polymer hybrids by EGA and EGA-GC/MS (2)

[Background] Chitin is a natural polysaccharide found in the shell of crustaceans, cuticles of insects, etc. Chitin derivatives having poly(2-alkyl-2-oxazoline) sidechains with a high miscibility toward synthetic polymers have been blended with commodity polymers such as PVA and PVC in an attempt to yield new functional materials. Here, pyrolysis techniques such as EGA-MS, and Py-GC were applied to the characterization of a chitin-graft-poly(2-methyl-2-oxazoline)/PVA blend system.

[Experimental] Chitin-graft-poly(2-methyl-2-oxazoline) was prepared according to the established procedure, where living poly(2-methyl-2-oxazoline) side chains (degree of polymerization=19.6, Mw/Mn=1.1) was selectively grafted onto free amino groups of the partially N-deactivated chitin (degree of acetylation=52%). In the EGA-MS system used in this study a pyrolyzer (PY-2020D, Frontier Lab) attached to the injector of a GC was directly coupled with a quadrupole MS via a deactivated stainless steel capillary transfer-line.

[Results] The dependence of the blending ratio on the degradation behavior of the PVA moiety in the blend system was studied by EGA-MS in a SIM mode. As in Fig. 1(a), water derived from the dehydration at 260ºC is preceded by the evolution of 2-butenal at 270ºC. On the other hand, as in Fig. 1(b) both of the evolution profiles for water and 2-butenal for the B(60/40) blend sample shifted to higher temperatures by 50ºC than those for PVA alone. It is noted that similar evolution profiles in the region below 290ºC suggest that dehydration and scission of the main chain of PVA in the blend take place almost at the same time. Figure 2 show the relationship between the contents of the chitin derivative and the relative amounts of the representative pyrolysis products obtained by Py-GC at 600ºC. The relative yields of 2-butenal produced from PVA increased by blending with chitin derivative. This may be due to the lowered crystallinity of PVA in the blend system, resulting dense intermolecular interactions, thus leading to the main chain cleavages of PVA. The relative amount of acetamide and N-ethylacetamide decreased by blending with PVA. This suggests that the PVA molecules in the blend system interact with the whole molecules of the chitin derivatives and affect the degradation.

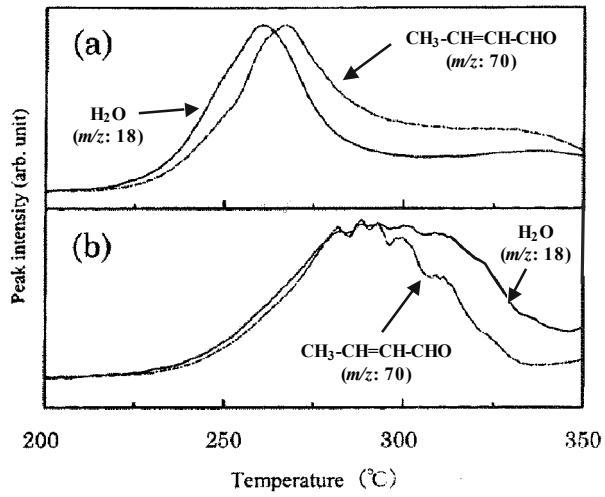

Figure 1. Selected ion monitoring (SIM) curves for water (m/z=18) and 2-butenal (m/z=70) at the first degradation stage between 200 and 350°C of a (a) PVA and (b) B(60/40) blend sample measured by EGA-MS. Peak heights are normalized.

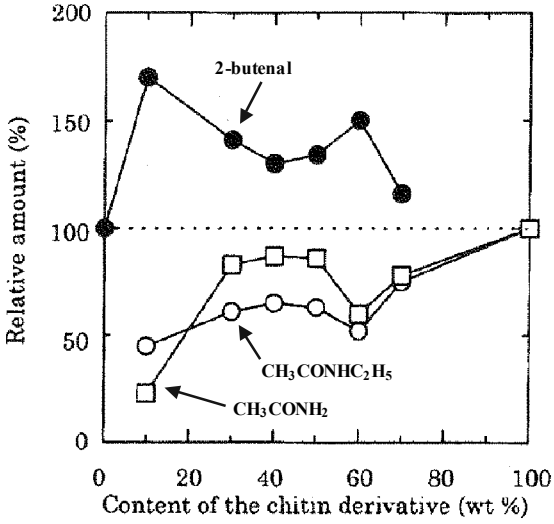

Figure 2. Relationship between composition of the blend and relative amounts of evolved 2-butenal, acetamide, and N-ethylacetamide obtained by Py-GC. Broken line indicates hypothetical evolution with no intermolecular interactions.

*Contents excerpted from H. Sato, H. Ohtani, S. Tsuge, K. Aoi, A. Takasu, M. Okada, *Macromolecules* 2000, 33, 357-362

Key word : EGA-MS, Py-GC, Chitin, PVA, Thermal degradation, SIM, 2-Butenal, Acetamide

Applications : General polymer analysis

Related technical notes : PYA3-008E

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R&D and manufactured by :

Frontier Laboratories Ltd.

1-8-14 Saikon, Koriyama
Fukushima-ken 963-8862 JAPAN
Phone: (81)24-935-5100 Fax: (81)24-935-5102

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