Characterization of chitin-based polymer hybrids by EGA and EGA-GC/MS (1)

[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, EGA-GC/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] Figure 1 shows evolution profiles of the thermal degradation products from the chitin derivative/PVA blends measured by EGA-MS as function of programmed temperature together with those for the chitin derivative and PVA each. The TIC curve of PVA shows two-stage degradation. The thermal degradation of the blends samples also occurs in two stages, reflecting the degradation of both constituent polymers. The thermal degradation products were identified by EGA-GC/MS. Figure 2 shows the TIC chromatograms of (a) the evolved products cold-trapped during the first degradation stage from 240 to 340°C and (b) those during the second stage from 340 to 480°C for the B(60/40) blend sample. The major products such as water and various unsaturated and aromatic aldehydes as shown Fig. 2a may be formed during the first degradation stage through dehydration of PVA followed by scission of the resulting polyene chains. On the other hand, various degradation products originating from the chitin derivatives are formed during the second degradation stage as shown in Fig. 2b. Among the products were acetamide from the chitin main chain, and acetone and other nitrogen containing compounds from scission and/or rearrangement in polyoxazoline side chains.

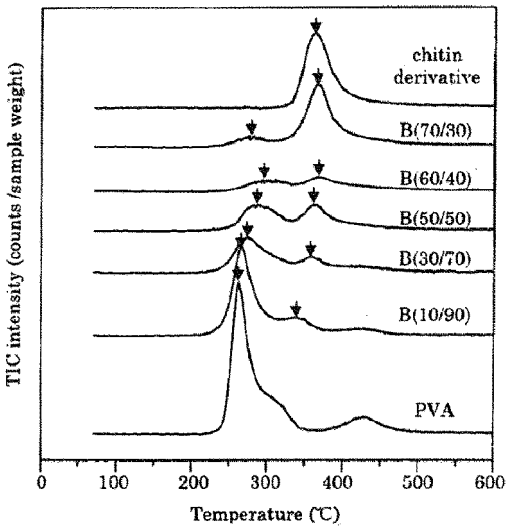

Figure 1. Evolution profiles of the thermal degradation products from the chitin derivative/PVA blend samples observed in TIC by EGA-MS.

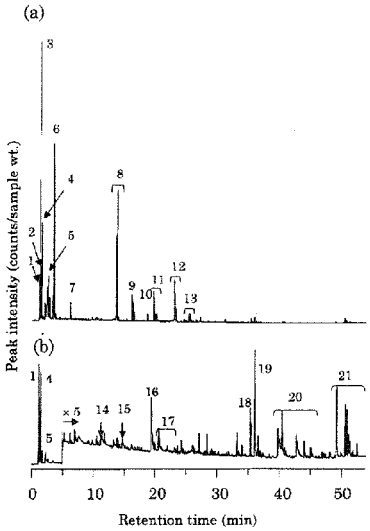

Figure 2. TIC chromatograms of the degradation products formed from the B(60/40) blend during heating ranges (a) 240-340°C and (b) 340-380°C.

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