

Structural Characterization of Hydrogenated Acrylonitrile-Butadiene Rubbers by Py-GC

[Background] Acrylonitrile-butadiene rubbers (NBRs) have their high oil-resistant properties and are used in a variety of applications. However, NBRs have relatively low thermal stability due to the presence of double bonds from butadiene (BD). Hence, hydrogenation is required to improve the thermal stability. It is therefore important to characterize their microstructures and hydrogenation mechanism.

[Experimental] Hydrogenated NBRs were prepared by dissolving NBR in THF followed by hydrogenation in the presence of Pd catalyst. The high-resolution Py-GC system had a vertical micro-furnace and was directly attached to a GC with an FID. About 70µg each of samples was pyrolyzed at 550°C under nitrogen carrier gas (50mL/min). The identifications of peaks were done by a directly coupled GC-MS with both EI and CI sources.

[Results] Figure 1 shows the pyrograms of NBR samples at 550°C before and after the hydrogenation that were obtained using a fused silica capillary column with poly(dimethylsiloxane) stationary phase. Characteristic peaks in the pyrogram of N-37(0) were butadiene (BD) monomer, BD dimer, and acrylonitrile (AN) monomer; whereas those of hydrogenated NBR consisted of a series of linear mononitriles (MN(A)s) up to C₁₂, each of which consisted of a doublet corresponding to an α-olefinic MN(A) (the former) and a saturated MN(A) (the latter). Another series of mononitrile positional isomers (MN(B)s) are also observed. HC peaks of each carbon number consisted of a triplet corresponding to to an α,ω-diolefin, and α-olefin, and a n-alkane.

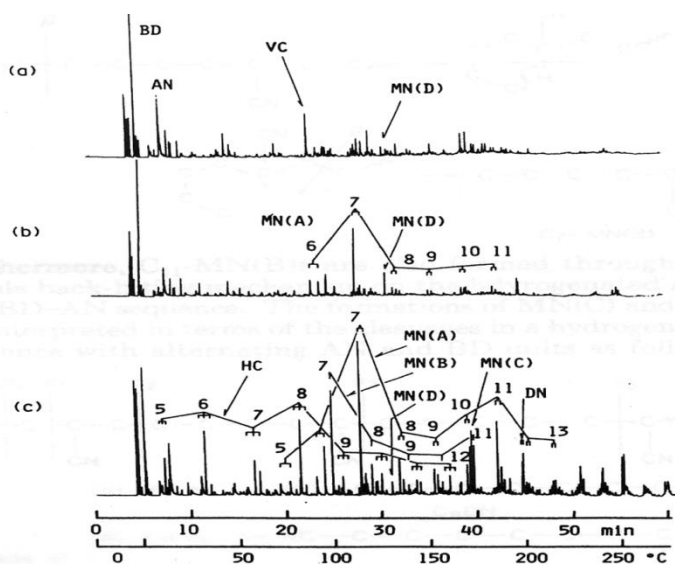


Figure 1. Pyrograms of NBRs before and after hydrogenation at 550°C separated by a poly(dimethylsiloxane) column: (a) N-37(0); (b) N-37(44); (c) N-37(98). See Table 1 for abbreviations. Numbers indicate carbon numbers of compounds.

Table 1 Characteristic Degradation Products from Hydrogenated NBR

compound class	abbreviation	sequence
Butadiene	BD	B
Butadiene dimer (4vinylcyclohexane)	VC	BB
Acrylonitrile	AN	A
Hydrocarbons	HC	EE EEE
Mononitriles	MN(A)	EA
	MN(B)	EEEA EA
	MN(C)	EEA
	MN(D)	EAE BA
Dinitriles	DN	AEA

B = 1,4-butadiene unit; A = acrylonitrile; E = hydrogenated 1,4-butadiene unit

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Keywords : NBR, Py-GC, Hydrogenation, Pyrogram

Products used : Multi-functional pyrolyzer

Applications : General polymer analysis

Related technical notes :

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