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APPENDIX

APPENDIX A

GRAFT COPOLYMERIZATION PARAMETERS (1)

Graft copolymerization parameters, used for studying the reaction condition of graft copolymerization, are shown in the following:

1. Conversion of Monomer to Polymer

Conversion of monomer to copolymer was used to describe the reaction yield. It represented the total amount of polymer formed, including both homopolymer and grafted polymer, with respected to the amount of monomer charged. It was calculated by:

$$\% \text{ Conversion of monomer to polymer} = \{[A - B]/C\} \times 100$$

when; A = dry weight of products isolated by filtration, after the reaction has been terminated (before extraction), (g),

B = dry weight of microcrystalline cellulose (g),

C = weight of monomer (g)

2. Conversion of Monomer to Copolymer

Conversion of monomer to copolymer represented the amount of polymer chemically bounded to the microcrystalline cellulose (i.e., not removable by extraction), with respected to the amount of monomer charged. It was calculated by:

$$\% \text{ Conversion of monomer to copolymer} = \{[D - B]/C\}x100$$

when; D = dry weight of product after polymerization and extraction

3. Homopolymer

The analysis of the homopolymer content was made by the extraction with N,N-dimethylformamide(DMF). It was measured as the decrease in weight of the product by extraction. DMF is a good solvent for polyacrylonitrile and polyacrylic acid homopolymer but a poor solvent for the graft copolymer.

4. Add-On

Add-on represented the synthetic polymer in the graft copolymer. The calculation is:

$$\% \text{ Add-on} = \{[D-B]/D\}x100$$

5. Grafting Ratio

Grafting ratio represented the weight ratio of polymer in graft per cellulose.

6. Grafting Efficiency

Grafting efficiency represented the total synthetic polymer formed that has been grafted to the microcrystalline cellulose. It calculation is:

$$\% \text{ Grafting efficiency} = \{[D-B]/[A-B]\}\text{x}100$$

7. Grafting Percentage

The grafting percentage was computed as the percent increase in the weight of grafted sample over the original weight of cellulose sample. It was calculated by:

$$\% \text{ Graft} = \text{Grafting percentage} = \{[D-B]/B\}\text{x}100$$

8. Graft Level

% Graft level was measured from the weight of grafted cellulose after homopolymer was extracted minus weight of cellulose sample, multiplied by 100.

APPENDIX B

INTERPRETATION OF THE IR SPECTRA OF THE RELATED COMPOUNDS (29)

1) CELLULOSE

Absorption frequency (cm^{-1})	Intensity	Remark and Assignment
3400-3500	S	O-H stretching of crystalline part and intramolecular H-bonds of cellulose
2900	Sh	C-H stretching
1400-1430	M	O-H in plane bending, "crystallinity band," CH_2 bending
1370-1380	M	C-H bending,
1310-1330	M	O-H in plane bending, CH_2 wagging
1280	W	C-H bending
1200-1240, 1250-1270	W	O-H in plane bending
1170	M	stretching of C-O in ring, bending of C-OH antisym., bridge oxygen stretching

Absorption frequency (cm^{-1})	Intensity	Remark and Assignment
1110-1120	S	antisym. in phase ring wagging
		C-O-C streching,
		"association band,"
		antisym. in phase ring-stretching
1060	S	OH bending
1010-1030	S	C-O stretching
990-1000	S	C-O stretching,C-C stretching
890-900	W	CH bending, characteristic of β linkage (ring stretching),
		CH_2 stretching,"amorphous-band,"
		antisym. out-of-phase-stretching
		ring breathing
670	M	OH out-of-plane bending

2) POLY(ACRYLONITRILE-CO-ACRYLIC ACID)

Absorption frequency (cm^{-1})	Intensity	Remark and Assignment
3100-3550	S	O-H stretching
2900-2980	M	C-H stretching of an aliphatic compound
2240	Sb	C≡N stretching
1710-1760	Sh	C=O stretching
1480	Sh	CH_2 bending
1395-1440	M	O-H in plane bending C-O stretching
1360	M	C-H bending
1320-1210	M	C-O stretching O-H in plane bending
1250	M	CH_2 bending

3) CYANOETHYLATED MICROCRYSTALLINE CELLULOSE

Absorption frequency (cm^{-1})	Intensity	Remark and Assignment
3400-3500	S	O-H stretching of crystalline part and intramolecular H-bonds of cellulose
2850-2980	Sh	C-H stretching
2240	Sh	C≡N stretching
1400-1430	M	O-H in plane bending, "crystallinity band," CH_2 bending
1360-1370	M	C-H bending
1310-1330	M	O-H in plane bending, CH_2 wagging
1200-1240,1250-1270	W	O-H in plane bending
1160	S	stretching of C-O in ring, bending of C-OH antisym., bridge oxygen stretching
1110-1120	S	antisym. in phase ring wagging C-O-C stretching, "association band," antisym. in phase ring stretching

Absorption frequency (cm^{-1})	Intensity	Remark and Assignment
1060-1150	S	C-O-C asym. stretching
1060	S	OH bending
1010-1030	S	C-O stretching
990-1000	S	C-O stretching, C-C stretching
850-860	W	C-H bending, characteristic of β linkage (ring stretching), CH_2 stretching, "amorphous band," antisym. out -of-phase stretching

4) MICROCRYSTALLINE CELLULOSE-GRAFT-POLYACRYLONITRILE

Absorption frequency (cm^{-1})	Intensity	Remark and Assignment
3300-3500	S	O-H stretching of crystalline part and intramolecular H-bonds of cellulose
2900	Sh	C-H stretching
2240	Sh	C≡N stretching
1440	Sh	CH_2 bending
1370-1380	M	C-H bending
1310-1330	M	O-H in plane bending, CH_2 wagging
1280	W	C-H bending
1200-1240,1250-1270	W	O-H in plane bending
1160	S	stretching of C-O in ring, bending of C-OH antisym., bridge oxygen stretching
1110-1120	S	C-O-C stretching
1060	S	OH bending
1010-1030	S	C-O stretching
990-1000	S	C-O stretching, C-C stretching
890-900	W	CH bending, characteristic of β linkage

5) MICROCRYSTALLINE CELLULOSE - GRAFT - POLY(ACRYLONITRILE - CO - ACRYLIC ACID)

Absorption frequency (cm^{-1})	Intensity	Remark and Assignment
3300-3550	S	O-H stretching of crystalline part and intramolecular H-bonds of cellulose, O-H stretching of polyacrylic acid
2900-2980	M	C-H stretching of an aliphatic compound
2240	Sh	C≡N stretching
1710-1760	M	C=O stretching
1480	Sh	CH_2 bending
1400-1430	M	O-H in plane bending "crystallinity band"
1395-1440	M	O-H in plane bending, C-O stretching
1360	M	C-H bending
1310-1330	W	O-H in plane bending, CH_2 wagging

Absorption frequency (cm^{-1})	Intensity	Remark and Assignment
1280	W	C-H bending
1200-1240,1250-1270	W	O-H in plane bending
1170	M	stretching of C-O in ring, bending of C-OH antisym., bridge oxygen stretching
1110-1120	S	C-O-C stretching
1060	S	OH bending
1010-1030	S	C-O stretching
990-1000	S	C-O stretching, C-C stretching
890-900	W	CH bending, characteristic of β linkage

6) WATER-RETAINING MATERIAL

Absorption frequency (cm^{-1})	Intensity	Remark and Assignment
3200-3500	S	N-H stretching
2880-2980	W	C-H stretching of an aliphatic compound
1670	S	C=O stretching
1530-1620	S	N-H bending
1550	S	asym. carboxylate anion $C(\quad O)_2$ stretching
1425	M	C-N stretching
1385-1400	M	sym. carboxylate anion $C(\quad O)_2$ stretching
800-666	M	out-of-plane NH wagging

7) SODIUM ALLYL SULFONATE MONOMER

Absorption frequency (cm^{-1})	Intensity	Remark and Assignment
3060-3080	W	CH_2 asym. stretching
2980-2900	W	CH_2 sym. stretching
1600-1650	M	C=C stretching vibrations of an alkene
1410-1420	M	$=CH_2$ in-plane bending
1150-1260	S	SO_2 asym. stretching
1048	S	SO_2 sym. stretching
985,910	Sh	$=CH_2$ out-of-plane bending
650	Sh	S-O stretching

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