ORIGINAL**Organic Builders. XXIII.*¹ Preparation and Builder Performance of Poly[(sodium acrylate)-*co*-(2-cyclohexen-1-one)]**

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Poly[(sodium acrylate)-*co*-(2-cyclohexen-1-one)]s containing hydrophobic cyclohexanone rings and enzymatically active carbonyl groups were prepared and their building performances in detergents were examined. Preparations were made by the radical polymerization of 2-cyclohexen-1-one with methyl acrylate or acrylic acid. Detergency tests were carried out using heavy duty detergent formulations on the soiled cotton cloths. It was found that these copolymers showed a better detergency building performance than those of trisodium citrate and zeolite with some of them superior to those of sodium tripolyphosphate (STPP), and also showed an improved biodegradability. It seems that the cyclohexanone ring in the poly(carboxylic acid) acts as a booster of detergency as well as a biodegradation unit.

1 Introduction

Polymeric polycarboxylates containing carbon-carbon backbones and a relating high charge density of carboxylate groups along the chain have been investigated for STPP substitutes and a number of polyelectrolytes, such as poly(sodium acrylate), have been reported to give excellent builder performance when compared with STPP on an equal weight basis^{1)–4)}. These polymeric compounds, however, are extremely resistant to biodegradation³⁾, which is an important criterion for an environmentally acceptable compound as a STPP replacement⁴⁾. Therefore, a molecular design to develop biodegradable polymeric carboxylates containing carbon-carbon backbones are needed. The most important property of a detergent builder is probably the sequestering properties for Ca ions in washing solutions. It has been reported that the polycarboxylates can coil back on themselves in solution as they form complexes with Ca creating low energy intramolecular binding sites which are probably less dependent on specific molecular geometry than specific binding sites of lower molecular weight compounds. Thus, a specific

polymer structure will be less important to functionality²⁾. It seems that the incorporation of a small amount of biodegradable segment to a polycarboxylate chain will improve its biodegradability without lowering its builder performances. We recently reported that the introduction of hydroxyl groups or ether groups into the polycarboxylate chain improved their biodegradability^{5)–8)}.

In this report, poly[(sodium acrylate)-*co*-(2-cyclohexen-1-one)] containing a cyclohexanone ring as a biodegradable segment as well as a booster of detergency was prepared. Its biodegradability and its builder performance in detergent were examined.

2 Experiments

2.1 Materials and measurements

All materials were of the highest available purity and used as purchased. Poly(sodium acrylate)s having number-average molecular weights, $\overline{M}_n$, of 9020 and 79200, and molecular weight distributions, $\overline{M}_w/\overline{M}_n$, of 1.8 and 1.1, respectively, were obtained from the Aldrich Chemical Co, Inc. Molecular weights and molecular weight distributions were measured in 0.1 M phosphate buffer (+0.3 M sodium chloride, pH 6.8) on a JASCO gel permeation chro-

*1 Organic Builders. XXII. See ref. 8).

matographic system (GPC) with commercial GPC columns (TSK-GEL G 5000 PW+G 2500 PW, TOSO Co. Ltd.), calibrated with a poly (ethylene glycol) standard.

2.2 Preparation of poly[(sodium acrylate)-*co*-(2-cyclohexen-1-one)] [P(SA-CH-O)] obtained from the polymerization of methyl acrylate with 2-cyclohexen-1-one

P(SA-CHO) was prepared by radical polymerization of 2-cyclohexen-1-one with methyl acrylate followed by alkaline hydrolysis, as shown in **Scheme-1**. Preparation of P(SA-CHO) having a molecular weight of 51500 and sodium acrylate content of 94 mol % [P(SA-CHO)-51500 (94)] is described as a typical example. A mixture of methyl acrylate (10.0 g, 0.132 mol), 2-cyclohexen-1-one (2.24 g, 0.023 mol) and benzoyl peroxide (0.33 g, 0.0014 mol) was stirred at 70°C for 24 h in a glass autoclave with an argon atmosphere. After the reaction, the crude polymer was dissolved in a small amount of acetone (20 mL) and

the solution was then added slowly to a large amount of ether (200 mL) with vigorous stirring to precipitate the polymer. This purification procedure was repeated three times to obtain 7 g of poly[(methyl acrylate)-*co*-(2-cyclohexen-1-one)] in 57.2 % yield.

FT-IR : 1735 (COOMe), 1670 (C=O), 2951, 1450 cm⁻¹ (CH₂). ¹H-NMR (90 MHz : CDCl₃) : δ = 1.3~1.8 (CH₂ of methyl acrylate unit, CH₂ at C 4 and C 5 of cyclohexanone unit), 2.2~2.6 (>CH-, and -CH₂CO-), 3.7 (COOMe).

The content of the methyl acrylate fraction in the copolymer determined by ¹H-NMR was 94 mol %. Poly [(methyl acrylate)-*co*-(2-cyclohexen-1-one)] (7.0 g) was saponified with an excess of 4 M sodium hydroxide (1.5 equivalent excess) with stirring at 110°C for 24 h in an autoclave. The crude product was purified by dissolution in a minimum amount of distilled water, reprecipitated with ethanol, and dialyzed exhaustively against distilled water for a week, to give P(SA-CHO)-51500 (94) almost quantita-

Scheme-1 Preparation of poly[(sodium acrylate)-*co*-(2-cyclohexen-1-one)].

Table-1 Polymerization condition and analytical data of poly[(sodium acrylate)-*co*-(2-cyclohexen-1-one)]

Code	Polymerization condition					Polymer (sodium salt)			
	M 1	M 2	Molar ratio [M 1]/[M 2]	Temp. (°C)	Time (h)	Yield (%)	M _n	M _w /M _n	Content of M 1 (mol%)
P(SA-CHO)-									
51500(94)	MeA	CHO	5.0	70	24	53	51500	1.6	94
30900(93)	MeA	CHO	3.0	70	24	62	30900	1.6	93
17100(94)	MeA	CHO	1.0	70	24	45	17100	1.4	94
11800(81)	MeA	CHO	0.5	90	24	20	11800	1.7	81
10900(79)	MeA	CHO	0.25	90	24	4	10900	1.7	79
P(SA-CHO)A-									
33600(83)	AA	CHO	4.0	90	20	48	33600	2.4	83
24500(81)	AA	CHO	3.0	90	20	48	24500	2.4	81
19300(80)	AA	CHO	1.0	90	20	42	19300	1.7	80
9890(72)	AA	CHO	0.5	90	24	29	9890	2.0	72
7310(75)	AA	CHO	0.25	90	24	11	7310	1.8	75
4620(62)	AA	CHO	0.2	110	24	8	4620	1.9	62

tively as a powder. $\overline{M}_n=51500$ and $\overline{M}_w/\overline{M}_n=1.6$ by GPC. FT-IR : 1570 (COONa), 1697 (C=O), 2950, 1454 cm^{-1} (CH_2). ^{13}C -NMR (22.5 MHz : D_2O) : $\delta=36.7\sim39.4$ (CH_2 of sodium acrylate, CH_2 at C 4 and the C 5 of cyclohexanone unit), 45.8~47.6 ($>\text{CH}-$), 185.5 (COONa).

Polymer code, polymerization condition and analytical data are summarized in Table-1.

2-3 Poly[(sodium acrylate)-*co*-(2-cyclohexen-1-one)] obtained from the polymerization of acrylic acid with 2-cyclohexen-1-one P(SA-CHO)A

P(SA-CHO)A was prepared by the polymerization of acrylic acid with 2-cyclohexen-1-one followed by alkaline saponification, shown in Scheme-1. Preparation of P(SA-CHO)A having a molecular weight of 24500 and a sodium acrylate content of 81 mol % [P(SA-CHO)A-24500 (81)] is described as a typical example. A mixture of acrylic acid (9.6 g, 0.133 mol), 2-cyclohexen-1-one (3.2 g, 0.033 mol) and benzoyl peroxide (0.32 g, 0.0013 mol) was stirred at 90°C for 20 h in a glass autoclave with an argon atmosphere. After the reaction was over, the crude polymer was dissolved in a small amount of methanol (50 mL) and the solution was then added slowly to a large amount of ether (200 mL) with vigorous stirring to precipitate the polymer. This procedure was repeated three times to obtain poly[(acrylic acid)-*co*-(2-cyclohexen-1-one)] in 41 % yield. The content of acrylic acid fraction in the copolymer was determined by the titration method. Copolymer (100 mg) and an excess amount (10 mL) of 0.5 N potassium hydroxide was refluxed for 3 h. The excess amount of potassium hydroxide was then calculated by the titration with 0.1 N hydrochloric acid, to determine the amount of acrylic acid fraction in the copolymer. Thus determined acrylic acid content was 81 mol %.

Poly[(acrylic acid)-*co*-(2-cyclohexen-1-one)] (5.5 g) was neutralized with an excess of 4 M sodium hydroxide (1.5 equivalent excess) at 100°C for 2 h with stirring. The crude sodium salt of the copolymer was purified by dissolution in 40 mL of distilled water, reprecipitated with 200 mL of ethanol, and dialyzed exhaustively against distilled water for a week to give P(SA-CHO)A-24500 (81) almost quantitatively, as a white powder. $\overline{M}_n=24500$ and $\overline{M}_w/\overline{M}_n=2.4$

by GPC.

FT-IR : 1572 (COONa), 1697 (C=O), 2948, 1454 cm^{-1} (CH_2). ^{13}C -NMR (22.5 MHz : D_2O) : 36.5~40.0 (CH_2 of sodium acrylate, CH_2 at C 4 and C 5 of the cyclohexenone unit), 45.8~47.6 ($>\text{CH}-$), 185.5 (COONa).

Polymer code, polymerization condition and analytical data are summarized in Table-1.

2-4 Detergency

The detergency test was first conducted with a standard heavy duty detergent formulation containing 20 % sodium dodecylbenzenesulfonate, 25 % STPP/ODA, 5 % sodium silicate, 3 % sodium carbonate, 0.5 % carboxymethyl cellulose (CMC) and 46.5 % sodium sulfate to determine the detergency of the STPP/ODA formulation as a basis for comparison with the test builders. In the experimental formulas the STPP was replaced with an equal weight of the test builders. The washing efficiency of the builders was determined in Terg-O-Tometer using improved artificially soiled cotton cloth test pieces prepared from an aqueous dispersion method⁹. The washing experiments were performed with water of 3° DH with a cloth to liquor ratio of 1 : 50, at a temperature of 25°C and with a detergent concentration of 1.2 g/L. Light reflectance of the test swatches was measured by means of an automatic reflectometer equipped with a green filter. The K/S ratio was calculated using the Kubelka-Munk equations¹⁰, and the detergency was expressed according to the equation as :

$$\text{Detergency (\%)} = (A - B) / (A - C) \times 100$$

where *A*, *B* and *C* are the K/S values of the soiled swatches, of the washed swatches, and the original unsoiled swatches, respectively.

$$K/S = (1 - R) / 2R \quad R : \text{reflectance}$$

Relative detergency was expressed as a value of 10 for STPP and 0 for disodium 3-oxapentanedioate (ODA).

2-5 Calcium sequestration capacity¹¹⁾

A calcium ion electrode (Model 93-20, Orion Research, Inc.) and an ion meter (IM-20 E, TOA Electronic Ltd.) were used to measure the equilibrium calcium ion concentrations. Builder (10 mg) was dissolved in 50 mL of 1.00×10^{-3} M, 40 g/L calcium hardness solution with a 0.08 M KCl (ion strength $\mu=0.08$), and the pH of the solution was adjusted to 9.0 at 30°C. The electrode was immersed in the solution and

stirred. After 10 min, equilibrium free calcium ion concentrations were measured, and the calcium sequestration capacity was expressed as grams of calcium ion sequestered by 100 g of the builder.

2.6 Dispersion capacity¹⁾

Dispersion capacity for manganese dioxide was measured according to the titration method¹²⁾ and expressed as mg of dispersed manganese dioxide in a 100 mL of 0.05 % aqueous builder solution after 4 h sedimentation at 25°C.

2.7 Biodegradation^{5)~7)}

The five-day biochemical oxygen demand (BOD₅) was determined by the oxygen consumption method according to the Japanese Industrial Standard (JIS K 0102) using activated sludge obtained from a municipal sewage plant. Residual polymers in the culture broth before and after the biodegradation test (BOD₅ test) were directly analyzed by GPC.

3 Results and Discussions

3.1 Preparation of polymers

Poly[(sodium acrylate)-*co*-(2-cyclohexen-1-one)]s were prepared by the polymerization of 2-cyclohexen-1-one with methyl acrylate or acrylic acid. Copolymerization of 2-cyclohexen-1-one with acrylic acid/methyl acrylate does not seem to be efficient, since the reactivity ratios of the two monomers are much different, and the acrylate radical is too unreactive to add the unreactive 2-cyclohexen-1-one monomer. Thus, the resultant polymer contains a longer or shorter acrylate chain separated by a unit of 2-cyclohexen-1-one at a random position. As indicated in **Table-1**, 2-cyclohexen-1-one fractions in the resultant polymer were slightly higher when acrylic acid was used as a comonomer instead of methyl acrylate. In both cases, as the monomer

fractions of cyclohexen-1-one was increased in the polymerization reaction, the content of 2-cyclohexen-1-one in the resultant polymer was also increased, but the polymer yield was significantly lowered. The P(SA-CHO) and P(SA-CHO)A showed no significant differences in both physico-chemical properties and biological properties, suggesting that the polymerization sequence of the two monomers is similar.

3.2 Detergency building performance

Detergency building performances of poly[(sodium acrylate)-*co*-(2-cyclohexen-1-one)]s were examined using a heavy duty detergent formulation on the soiled cotton cloths. Relative detergency expressed as a value of 10 for STPP and 0 for disodium 3-oxapentanedioate (ODA) is shown in **Fig. -1**. Poly[(sodium acrylate)-*co*-(2-cyclohexen-1-one)] showed better detergency building performances than those of ODA and trisodium citrate with some of them superior to those of STPP. The difference in molecular weight and contents of the 2-cyclohexen-1-one unit in detergency was not clearly recognized. This fact will be favorable for industrial applications. It was also found that the difference between building performance of P(SA-CHO) obtained from methyl acrylate and P(SA-CHO)A obtained from acrylic acid was not clearly seen, suggesting both polymers obtained by the two kinds of monomer sources are almost chemically identical. **Fig. -2** shows the detergency building performances of poly(sodium acrylate)s with the number-average molecular weights of $\overline{M}_n=9020$ and of $\overline{M}_n=79200$, and P(SA-CHO)-51500 (94) as well as STPP and ODA. It was found that the P(SA-CHO)-51500 (94) showed a better building performance than those of the poly(sodium acrylate)s. The introduction of a 2-cyclohexen-1-one unit to the poly(sodium acrylate) chain

Fig. -1 Detergency building performances of poly[(sodium acrylate)-*co*-(2-cyclohexen-1-one)] , STPP, ODA, trisodium citrate and zeolite.

Fig. -2 Detergency building performances of P(SA-CHO), poly(sodium acrylate)(PSA), STPP and ODA.

improved their detergency. It will be anticipated that the 2-cyclohexen-1-one moiety of P(SA-CHO) will act as a booster of detergency by the hydrophobic effect, and the absorption of P(SA-CHO) onto lipophilic soils will be enhanced when compared with that of the polyacrylate homopolymer. The redeposition will then be inhibited by the electric repulsions during the washing process. Details are currently under investigation.

In attempting to mimic the various desirable properties of STPP, its chelating properties for calcium ion and magnesium ion, and dispersion capacities for soiled particles are probably the

most indispensable and are easily replaced by other substances^{(2), (13), (14)}. For this reason chelating capacities and dispersion capacities for MnO_2 as a model of polarized soils were measured and summarized in **Table-2**. It was found that these polycarboxylates showed excellent dispersion capacity and calcium sequestration capacity, better than those of ODA and sodium citrate⁽¹⁵⁾. A correlation between sequestering capacity for Ca and dispersion capacity for MnO_2 and resulting detergency performance was demonstrated by these data. These excellent effects are attributed to the relatively high charge density of carboxylate groups along the polymer chain.

3-3 Biodegradation

Table-3 shows the five-day biochemical oxygen demand (BOD_5) of poly[(sodium acrylate)-*co*-(2-cyclohexen-1-one)] and poly(sodium acrylate) having a number-average molecular weight of 8730. The BOD test is only an indication of susceptibility of products to biodegradation, but the test is often a useful tool for rapid screening. As shown in **Table-3**, the BOD_5 values of P(SA-CHO) and P(SA-CHO) A were low, but were higher than that of the corresponding poly(acrylic acid) (PSA). It seems that the introduction of a carbonyl group or 2-cyclohexen-1-one group to the polymer chain improves their biodegradability. It will be useful to measure the molecular-weight distributions of the polymers by GPC before and after biodegradation, whether the main chain of the polymer will be cleaved by microbes. From the GPC profiles of the cell-free filtrate of the biodegradation

Table-2 Calcium sequestration capacity and dispersion capacity of poly[(sodium acrylate)-*co*-(2-cyclohexen-1-one)], ODA and STPP.

Code	Sequestration capacity (g Ca/100 g sample 30°C, $\mu=0.08$, pH=9.0)	Dispersion capacity (mg of MnO_2 /100 mL of 0.05% soln.)
P(SA-CHO)-51500(94)	12.3	226
P(SA-CHO)-30900(93)	12.9	202
P(SA-CHO)-11800(81)	11.0	200
P(SA-CHO)-10900(79)	10.8	210
P(SA-CHO)A-24500(81)	12.5	126
P(SA-CHO)A-19300(80)	10.9	180
P(SA-CHO)A-9890(72)	11.2	186
P(SA-CHO)A-7310(75)	10.6	190
ODA	10.4	0
STPP	15.2	250

Table-3 Five-day biochemical oxygen demand (BOD_5) by oxygen consumption test (JIS K 0102)

Compound	ThOD (mg O/g)	BOD_5 (mg O/g)	$BOD_5/ThOD$ (%)
P(SA-CHO)-51500(94)	1030	61	5.9
P(SA-CHO)-30900(93)	1046	139	13.3
P(SA-CHO)-10900(79)	1265	142	11.2
P(SA-CHO)A-9890(72)	1374	200	14.6
P(SA-CHO)A-7310(75)	1327	176	13.3
P(SA-CHO)A-4620(62)	1530	49	3.2
PSA-9020	935	0	0

ThOD : Theoretical oxygen demand

PSA-9020 : Poly(sodium acrylate), $\bar{M}_n=9020$, $\bar{M}_w/\bar{M}_n=1.8$

(1) PSA $\bar{M}_n=9020$, (2) P(SA-CHO)-51500(94),
 (3) P(SA-CHO)-10900(79), (4) P(SA-CHO)A-33600(83),
 (5) P(SA-CHO)A-9890(72), (6) P(SA-CHO)A-4620(62)

— : 0 d, ---- : 5 d.

RI : Refractive index detection.

Fig. -3 GPC profiles of PSA and P(SA-CHO) before and after the biodegradation test (BOD_5) according to JIS K 0102.

media of poly(carboxylic acid) before and after the biodegradation test (BOD_5 test) according to JIS K 0102 shown in **Fig. -3**, it was found that the amount of polymer containing a biodegradable unit decreased after five days incubation and the biodegradation of the polymer undoubtedly occurred even if the BOD_5 value was low. High-molecular-weight fractions degraded equally as

well as the low-molecular-weight fractions, and substrate specificity towards molecular weight was not clearly recognizable. It was also found that the BOD_5 values and GPC profiles of P(SA-CHO) after the five days biodegradation test varied according to the source of activated sludge and to the seasons of the year. It seems that the probability of appearance of the microbes utilizing these types of copolymers is not so high in the environment. Under these circumstances, it will be necessary to isolate the polymer-degrading microbes for later discussion. Details will be analyzed in a subsequent paper. On the other hand, GPC profiles of PSA ($\bar{M}_n=9020$) showed no significant changes after 5 d incubation.

4 Conclusion

Poly[(sodium acrylate)-*co*-(2-cyclohexanone)] containing a hydrophobic cyclohexanone ring and an enzymatically active carbonyl group showed an improved biodegradability as well as building performances in detergents. It was found that the cyclohexanone ring in the poly(carboxylic acid) acted as a booster of detergency and a biodegradation unit.

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有機ビルダー(第23報)

ポリ[(アクリル酸ナトリウム)-*co*-(2-シクロヘキセン-1-オン)]の合成とビルダー性能

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疎水性のシクロヘキサノン環及び酵素反応を受けやすいカルボニル基を含有する一連のポリ[(アクリル酸ナトリウム)-*co*-(2-シクロヘキセン-1-オン)]を合成し、合成洗剤用ビルダー性能について検討を行った。ポリマーの合成は2-シクロヘキセン-1-オンとアクリル酸メチル又はアクリル酸とのラジカル共重合により行った。洗浄試験は木綿人工汚染布を用い、重質洗剤組成により行った。その結果、これらポリマーはクエン酸三ナトリウムやゼオライトよりもすぐれたビルダー性能を示し、一部のものはトリポリリン酸ナトリウム(STPP)よりもすぐれた性能を示した。さらに、これらポリマーは生分解性を有することが認められた。ポリ(アクリル酸ナトリウム)中におけるシクロヘキサノン環は洗浄力増強作用と生分解における活性部分としての両方の働きをしていることが予想された。

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第一線で活躍中の方々の手で「石油辞典」が刊行された。読者が目を通されるとその網羅する範囲の実に広いことに気付かれるであろう。基本的な化学物質から始まり、石油精製プロセス、石油製品、石油留分の性状関連用語とそれらの試験分析法、さらには石油化学や石油開発(探掘)に加え石油の輸送に至るまで、すべてカバーされており、約4,000の用語が収録されている。石油に関する知識はまさに「これ一冊あれば事足りる」ということになる。

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