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Silicon-Containing Condensation Polymers Derived from Long-Chain Fatty Acids

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Silicon-Containing Condensation Polymers Derived from Long-Chain Fatty Acids

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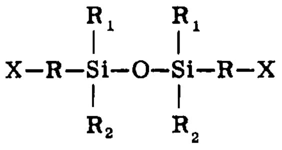
ABSTRACT

Silicon-containing condensation polymers were prepared starting from difunctional silicon-containing carboxylic acids or diols derived from long-chain fatty acids. Polyesters were synthesized by reaction of diacids containing siloxane linkages with diols, and diols containing siloxane linkages with various diacids. 1,3-Bis(10-carboxydecyl)tetramethyldisiloxane was condensed with various diamines to yield polyamides. 1,3-Bis(11-hydroxyundecyl)tetramethyldisiloxane and 1,3-bis[9(10)-hydroxyoctadecyl]tetramethyldisiloxane were condensed with diisocyanates to yield polyurethanes. Polycarbonates were synthesized by reacting the diols containing siloxane linkages with ethyl chloroformate. Some polybenzimidazols were synthesized by heating the diesters of the silicon-containing carboxylic acids with 3,3'-diaminobenzidine. Some of the polyurethanes and polybenzimidazoles were thermally stable up to 300°C as indicated by TGA.

INTRODUCTION

Some silicon-containing condensation polymers based on bis(p-carboxyphenyl)dimethylsilane and bis(hydroxymethylphenyl) dimethylsilane [1, 2] have been reported. Kovacs et al. [3] prepared some fully aromatic silicon-containing polymers which were stable at elevated temperatures.

We have recently synthesized [4] various difunctional disiloxane monomers derived from long-chain fatty acids:



$$X{-}R{-}\underset{R_2}{\overset{R_1}{Si}}{-}O{-}\underset{R_2}{\overset{R_1}{Si}}{-}R{-}X$$

$X = COOH, COOCH_3, CH_2OH$

$R = C_{10}H_{20}, C_{17}H_{34}$

$R_1R_2 = CH_3, C_6H_5$

These new difunctional disiloxane monomers were utilized as starting materials for the synthesis of some new condensation polymers containing long-chain alkyl fatty acid residues in their backbone. These new polymers may serve as a basis for increasing the industrial utilization of vegetable oils.

EXPERIMENTAL

Materials

Benzidine (May and Baker), p-phenylene diamine (B.D.H.), hexamethylene diamine (Fluka), ethylene-diamine (Riedel-De Haen), adipic acid (B.D.H.), sebacic acid (Fluka), succinic acid (B.D.H.), 2,4-toluene diisocyanate, 1,6-hexane diisocyanate (Fluka), 4,4'-diphenylmethane diisocyanate (B.D.H), 1,6-hexane diol (Aldrich), 1,2-ethane diol (Riedel-De Haen), and digol (Frutarom).

The silicon-containing compounds were prepared and purified as previously described [4].

Typical examples for the preparation of the various polymers are given below.

Polyester from 1,3-Bis[9(10)-hydroxyoctadecyl-tetramethyldisiloxane (VI) and Adipic Acid

A mixture of 1,3-bis[9(10)-hydroxyoctadecyl]tetramethyldisiloxane (1.34 g, 2.00 mmoles) and dried adipic acid (0.292 g, 2.00 mmoles) was heated in a glass tube under argon at atmospheric pressure to about 200° C. After 1 hr the reaction mixture was heated in vacuo (about 2 Torr) at 250° C for 4 hr. The viscous polymer was washed with methyl alcohol and dried under reduced pressure; the yield was 90%. It was soluble in benzene, carbon tetrachloride, and acetone. The intrinsic viscosity was determined in benzene at 25°C, $[\eta] = 0.09$ dl/g.

Analysis: Calculated for $C_{46}H_{92}Si_2O_5$: C, 70.77; H, 11.79. Found: C, 70.49; H, 11.05.

Several other polyesters were synthesized by the same procedure (Tables 1 and 2).

Determination of Molecular Weights of Polyesters

Number-average molecular weights were determined from the hydroxyl and carboxyl end groups of the polyesters [5, 6].

The carboxyl value is the weight in milligrams of NaOH required to neutralize the carboxyl end groups in 1 g of polymer. The hydroxyl value is the weight in milligrams of NaOH equivalent to the quantity of acetic acid used in the acetylation of hydroxyl groups present in 1 g of polymer.

Polyamide from 1,3-Bis(10-Carboxydecyl)tetramethyldisiloxane (III) and 1,6-Hexamethylene Diamine

Equivalent amounts of III and 1,6-hexamethylene diamine (0.004 mole) were heated in a glass tube under argon at atmospheric pressure to about 200°C. After 1 hr the reaction mixture was heated in vacuo at 230°C for 4 hr. The polyamide was obtained in 92% yield, m.p. 110°C. The polymer was purified by extraction with boiling methanol.

Analysis: Calculated for $C_{32}H_{66}Si_2O_3N_2$: C, 65.97; H, 11.34; N, 4.81. Found: C, 66.06; H, 11.16; N, 4.51.

Several other polyamides were synthesized by the same procedure (Table 3).

Determination of Molecular Weights of Polyamides

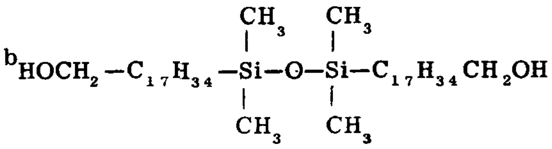
Number-average molecular weights were determined from the carboxyl and amine end groups of the polyamide [7]. Carboxyl end groups were determined by anhydrous titration of the polyamide (0.01 g) in DMSO or DMF (1 ml) with 0.1 N potassium methoxide in benzene/methanol using 0.2% thymol blue in dioxane as indicator, and amine end groups were determined by titration of the polymer (0.01 g) in acetic acid (1 ml) by 0.1 N perchloric acid in acetic acid using crystal violet 0.01% in acetic acid as indicator.

TABLE 1. Silicon-Containing Polyesters Derived from Oleic Acid[a]

Diol[b]	Diacid[b]	Yield (%)	COOH value,[c] mg NaOH/g polymer	OH value,[c] mg NaOH/g polymer	$(\eta)^d$ (dl/g)	$\overline{M}_n$[e]	$\overline{DP}_n$[e]	Carbon (%)		Hydrogen (%)	
								Calc	Found	Calc	Found
VI	Adipic	90	12.9	0.8	0.09	6000	7.7	70.77	70.49	11.79	11.05
VI	Sebacic	85	21.6	1.6	0.08	4000	5	71.71	70.86	11.96	11.47
VI	Succinic	95	8.0	0.8	0.08	9000	12	70.21	70.44	11.70	11.32
1,6-Hexane	VII	87	12.0	3.0	0.22	5335	7.1	70.80	68.28	11.79	11.20

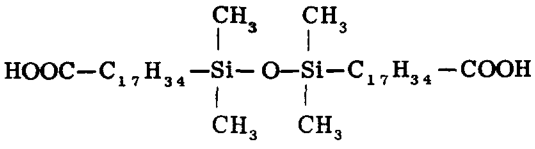
[a] Experimental conditions: Equivalent amounts of diacid and diol were heated at 200 to 250°C, for 4 hr.

[b]



$$HOCH_2-C_{17}H_{34}-\overset{\overset{CH_3}{|}}{\underset{\underset{CH_3}{|}}{Si}}-O-\overset{\overset{CH_3}{|}}{\underset{\underset{CH_3}{|}}{Si}}-C_{17}H_{34}CH_2OH$$

(VI)



$$HOOC-C_{17}H_{34}-\overset{\overset{CH_3}{|}}{\underset{\underset{CH_3}{|}}{Si}}-O-\overset{\overset{CH_3}{|}}{\underset{\underset{CH_3}{|}}{Si}}-C_{17}H_{34}-COOH$$

(VII)

[c] Determined by titration.

[d] The intrinsic viscosity was determined in benzene at 25°C.

[e] Calculated from end group content.

TABLE 2. Silicon-Containing Polyesters Derived from Undecenoic Acid[a]

Diol[b]	Diacid[b]	Yield (%)	COOH value,[c] mg NaOH/g polymer	OH value,[c] mg NaOH/g polymer	$(\eta)^d$ (dl/g)	$\overline{M}_n$[e]	$\overline{DP}_n$[e]	Carbon (%) Calc	Carbon (%) Found	Hydrogen (%) Calc	Hydrogen (%) Found
II	Adipic	94	6.0	5.2	0.21	7200	12.5	65.75	64.80	10.96	10.68
II	Sebacic	86	12.8	8.0	0.14	4000	6.5	67.50	66.36	11.25	11.15
II	Succinic	92	15.2	2.4	0.25	4600	8.3	64.75	63.69	10.79	10.74
II	III	79	8.8	5.6	0.09	5555	5.0	66.38	66.26	11.49	11.20
1,6-Hexane	III	92	5.8	5.4	0.19	7200	12.5	65.75	65.09	10.96	10.55
1,2-Ethane	III	98	10.4	8.8	0.09	4200	8.0	63.64	62.83	10.61	10.31
Digol	III	90	17.6	12.8	0.06	2700	5.0	64.75	65.03	10.79	10.57
IX	Adipic	88	11.2	6.4	0.10	4900	7.0	71.38	70.04	9.35	9.25
IX	Sebacic	89	9.6	8.0	0.22	4900	6.4	72.25	71.09	9.95	9.61
IX	Succinic	94	6.0	5.2	0.22	7200	10.6	70.59	69.65	9.41	9.15
1,6-Hexane	X	85	12.0	10.4	0.20	4000	5.7	71.18	70.04	9.60	9.42

[a] Experimental conditions: Equivalent amounts of diacid and diol were heated at 200 to 250 °C, for 4 hr.

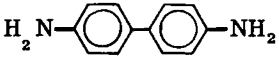
[b] $[HOCH_2-C_{10}H_{20}Si(CH_3)_2-]_2O$ (II) $[HOOC-C_{10}H_{20}Si(CH_3)_2-]_2O$ (III) $[HOCH_2-C_{10}H_{20}SiMePh-]_2O$ (IX) $[HOOC-C_{10}H_{20}SiMePh-]_2O$ (X)

[c] Determined by titration.

[d] The intrinsic viscosity was determined in benzene at 25° C.

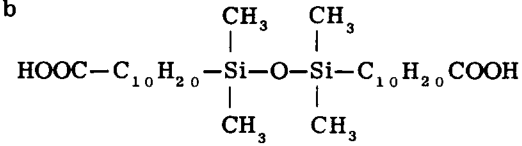
[e] Calculated from end group content.

TABLE 3. Silicon-Containing Polyamides Derived from Undecenoic Acid[a]

Diamine	Diacids[b]	Yield (%)	Melting point (°C)	End groups[c] (mmole/g × 10^{-3}) Amine	End groups[c] (mmole/g × 10^{-3}) Carboxyl	$[\eta]$[d] (dl/g)	$\overline{M}_n$[e]	$\overline{DP}_n$[e]	Carbon (%) Calc	Carbon (%) Found	Hydrogen (%) Calc	Hydrogen (%) Found
$H_2N-(CH_2)_6-NH_2$	III	92	110	4	0.2	0.12	5000	8.6	65.97	66.06	11.34	11.16
$H_2N-(CH_2)_2-NH_2$	III	80	130	4	0.3	0.11	4700	9.0	63.88	62.13	11.02	10.74
	III	89	148	1.2	1.2	0.17	8355	14.6	66.66	66.62	10.07	10.01
	III	84	195	0.6	0.5	0.18	18180	28.1	70.15	69.14	9.93	9.31

[a] Polymerization was carried out by heating equimolar amounts of the reactants at 200 to 240° C.

[b]



$$HOOC-C_{10}H_{20}-\underset{CH_3}{\overset{CH_3}{Si}}-O-\underset{CH_3}{\overset{CH_3}{Si}}-C_{10}H_{20}COOH$$

(III)

[c] Determined by titration.

[d] The intrinsic viscosity was determined in H_2SO_4 (96%) at 25° C.

[e] Calculated from end group content.

Polyurethane from 1,3-Bis[9(10)-hydroxyoctadecyl-tetramethyldisiloxane (VI) and 2,4-Toluene Diisocyanate

2,4-Toluene diisocyanate (0.348 g, 2.00 mmoles) and 1,3-bis[9(10)-hydroxyoctadecyl]tetramethyldisiloxane (VI) (1.35 g, 2.00 mmoles) were heated under argon so that the temperature reached 200°C after 30 min and the heating was continued for 3 hr. The polymer solidified on cooling and was washed with methyl alcohol, yield 95%, m.p. 30°C. The intrinsic viscosity was determined in 96% H_2SO_4 at 25°C, $[\eta] = 0.09$ dl/g.

Analysis: Calculated for $C_{49}H_{92}Si_2O_5N_2$: C, 69.67; H, 10.90. Found: C, 69.62; H, 10.78.

Several other polyurethanes were synthesized by the same proecdure (Table 4).

Polycarbonate from 1,3-Bis[9(10)-hydroxyoctadecyl-tetramethyldisiloxane (VI)

To a solution of 1,3-Bis[9(10)-hydroxyoctadecyl]tetramethyldisiloxane (VI) (2.01 g, 3.00 mmoles) in pyridine (10 ml), redistilled ethyl chloroformate (1.0 g, 9 mmoles) was added, and the mixture was stirred in the cold for 2.5 hr. Ice (8 g) was added to destroy excess ethyl chloroformate, the mixture was left overnight, and then it was extracted with ether. The ether layer was washed by dilute hydrochloric acid (7%) followed by water, dried over magnesium sulfate, and distilled in vacuo. A drop of titanium tetrachloride was added to the oily mixed-carbonate ester and was heated slowly to 200°C for 3 hr and at 260 to 280°C for 1 hr. The viscose polycarbonate was washed with methanol and then dried under reduced pressure over P_2O_5, yield 52%, $[\eta] = 0.05$ dl/g in benzene at 25°.

Analysis: Calculated for $C_{41}H_{84}Si_2O_4$: C, 70.69; H, 12.07. Found: C, 70.80; H, 11.98.

Two other polycarbonates were synthesized by the same procedure (Table 5).

Polybenzimidazole from 1,3-Bis(10-carbomethoxydecyl)tetramethyldisiloxane (I) and 3,3'-Diaminobenzidine

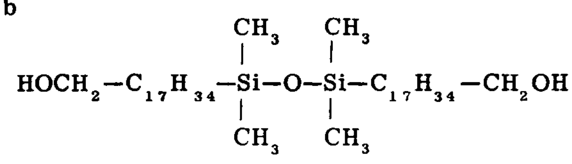
In a flask fitted with a nitrogen inlet tube were placed 1,3-bis(10-carbomethoxydecyl)tetramethyldisiloxane (I) (1.064 g, 2 mmoles) and 3,3'-diaminobenzidine (0.428 g, 2 mmoles). The reaction mixture was heated in a silicon oil bath under nitrogen to about 200 to 250°C

TABLE 4. Silicon-Containing Polyurethanes Derived from Long-Chain Fatty Acids[a]

Diol[b]	Diisocyanate	Melting point (°C)	Yield (%)	$[\eta]^c$ (dl/g)	Formula	Carbon (%)		Hydrogen (%)	
						Calc	Found	Calc	Found
VI	2,4-Toluene	30	95	0.09	$(C_{49}H_{92}Si_2O_5N_2)_n$	69.67	69.62	10.90	10.78
VI	1,6-Hexane	50-55	93	0.1	$(C_{48}H_{98}Si_2O_5N_2)_n$	68.74	70.52	11.70	11.58
VI	4,4'-Diphenylmethane	70-75	100	0.1	$(C_{55}H_{96}Si_2O_5N_2)_n$	71.74	72.88	10.43	9.94
II	2,4-Toluene	150-160	97	0.06	$(C_{35}H_{64}Si_2O_5N_2)_n$	64.81	64.41	9.88	9.60
II	1,6-Hexane	160-170	99	0.09	$(C_{34}H_{70}Si_2O_5N_2)_n$	63.55	62.20	10.90	10.48
II	4,4'-Diphenylmethane	190-200	96	0.1	$(C_{41}H_{88}Si_2O_5N_2)_n$	67.96	66.87	9.39	8.99

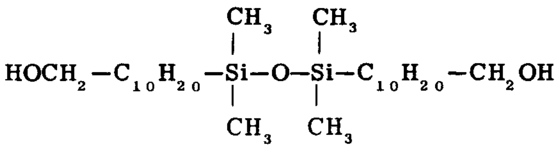
[a] Polyurethanes were synthesized by heating equivalent amounts of diols with diisocyanate at 200 to 240°C.

[b]



$HOCH_2-C_{17}H_{34}-Si(CH_3)_2-O-Si(CH_3)_2-C_{17}H_{34}-CH_2OH$

(VI)



$HOCH_2-C_{10}H_{20}-Si(CH_3)_2-O-Si(CH_3)_2-C_{10}H_{20}-CH_2OH$

(II)

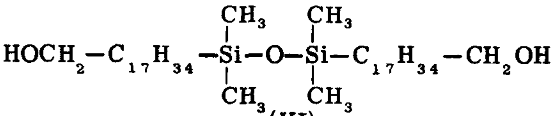
[d] The intrinsic viscosity was determined in H_2SO_4 (96%) at 25°C.

TABLE 5. Silicon-Containing Polycarbonates Derived from Long-Chain Fatty Acids[a]

Diol[b]	Yield (%)	$[\eta]^c$ (dl/g)	Formula	Carbon (%)		Hydrogen (%)	
				Calc	Found	Calc	Found
VI	52	0.05	$(C_{41}H_{84}Si_2O_4)_n$	70.69	70.80	12.07	11.98
II	58	0.21	$(C_{27}H_{56}Si_2O_4)_n$	64.80	62.27	11.20	10.58
IX	62	0.05	$(C_{37}H_{60}Si_2O_4)_n$	71.15	71.25	9.62	9.92

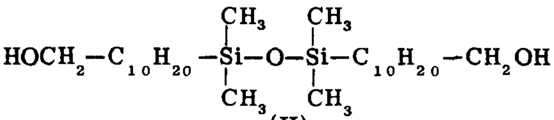
[a] The polycarbonates were synthesized by reacting diols with ethyl chloroformate in the presence of pyridine to give the mixed carbonate ester, which polymerized on heating in vacuo in the presence of $TiCl_4$.

[b]



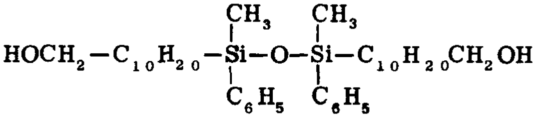
$$HOCH_2-C_{17}H_{34}-Si(CH_3)_2-O-Si(CH_3)_2-C_{17}H_{34}-CH_2OH$$

(VI)



$$HOCH_2-C_{10}H_{20}-Si(CH_3)_2-O-Si(CH_3)_2-C_{10}H_{20}-CH_2OH$$

(II)



$$HOCH_2-C_{10}H_{20}-Si(CH_3)(C_6H_5)-O-Si(CH_3)(C_6H_5)-C_{10}H_{20}CH_2OH$$

(IX)

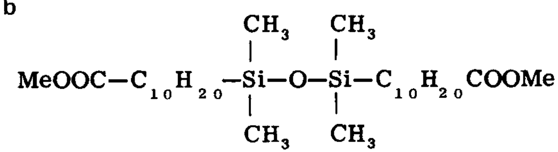
[c] The intrinsic viscosity was determined in benzene at 25° C.

TABLE 6. Silicon-Containing Polybenzimidazoles, Derived from Long-Chain Fatty Acids[a]

Diester[b]	Yield (%)	Melting point (°C)	$[\eta]$[c] (dl/g)	Formula	Carbon (%)[d] Calc	Carbon (%)[d] Found	Hydrogen (%) Calc	Hydrogen (%) Found
I	90	70	0.05	$(C_{38}H_{60}Si_2ON_4)_n$	70.81	70.82	9.32	9.36
VIII	92	110-130	0.1	$(C_{48}H_{64}Si_2ON_4)_n$	75.00	73.09	8.23	8.18
V	85	Viscose	0.05	$(C_{52}H_{88}Si_2ON_4)_n$	74.29	71.04	10.48	10.03

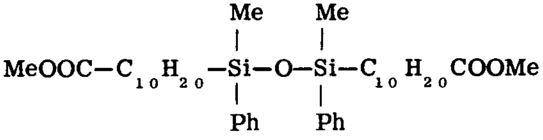
[a] Polybenzimidazoles were synthesized by heating equivalent amounts of diols with 3,3'-diaminobenzidine at 250 C.

[b]



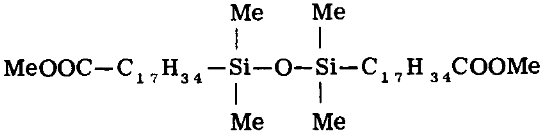
$MeOOC{-}C_{10}H_{20}{-}Si(CH_3)_2{-}O{-}Si(CH_3)_2{-}C_{10}H_{20}COOMe$

(I)



$MeOOC{-}C_{10}H_{20}{-}Si(Me)(Ph){-}O{-}Si(Me)(Ph){-}C_{10}H_{20}COOMe$

(VIII)



$MeOOC{-}C_{17}H_{34}{-}Si(Me)_2{-}O{-}Si(Me)_2{-}C_{17}H_{34}COOMe$

(V)

[c] The intrinsic viscosity was determined in H_2SO_4 (96%) at 25°C.

[d] Difficulties are often encountered in the analysis of polymers.

(1 hr) and then in vacuo (about 2 Torr) at 250°C for 4 hr. The polymer obtained was washed thoroughly with petroleum ether, yield 90%, m.p. 70°C. The intrinsic viscosity was determined in H_2SO_4 (96%) at 25°C, $[\eta] = 0.05$ dl/g.

Analysis: Calculated for $C_{38}H_{60}Si_2ON_4$: C, 70.81; H, 9.32. Found: C, 70.87; H, 9.36.

Other polybenzimidazoles were synthesized in the same manner (Table 6).

RESULTS AND DISCUSSION

For the synthesis of polyesters containing silicon, we investigated reactions of diacids containing siloxane linkages with diols, and diols containing siloxane linkages with various diacids. In general, the polyesters were prepared by heating equivalent amounts of diacids with diols (Tables 1 and 2). The reaction mixture was heated initially under atmospheric pressure (200°C) and then in vacuo (200 to 250°C.

The polyesters obtained were viscous. They were soluble in benzene, acetone, and solvents for polyesters. The number-average molecular weights were determined from the hydroxyl and carboxyl end groups of the polyesters [5, 6].

The carboxylic end groups were determined by direct titration of the polymer suspended in pyridine with aqueous (0.1 N) sodium hydroxide solution (carboxylic value). The hydroxylic end groups were determined by using pyridine-acetic anhydride acetylation reagent and titration with aqueous (0.1 N) sodium hydroxide solution (hydroxyl value).

The molecular weights were not high, and the polymers had a low inherent viscosity.

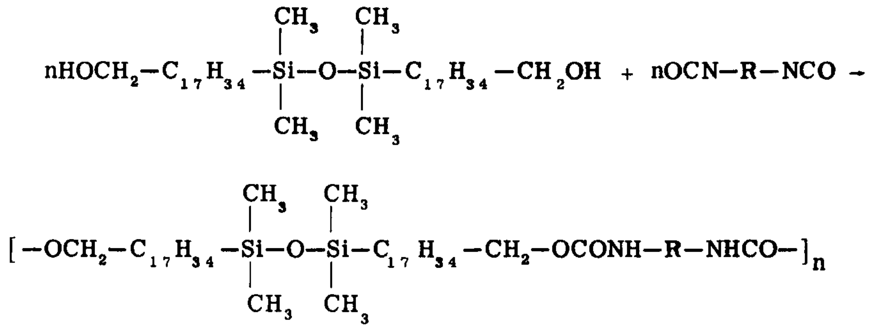
Some new silicon-containing polyamides were synthesized by reacting 1,3-bis(10-carboxydecyl)tetramethyldisiloxane (III), with diamines such as benzidine, p-phenylene diamine, hexamethylene diamine, and diethylene diamine.

In general, the polyamides were prepared by heating equivalent amounts of reactants. Initially the heating was conducted under atmospheric pressure and then in vacuo. The results are given in Table 3. The polyamides obtained from the more rigid diamines (benzidine and p-phenylene diamine) gave higher melting polymers than the corresponding polyamides obtained from hexamethylene diamine and ethylene diamine. The polyamides were insoluble in ordinary solvents such as chloroform, acetone, and benzene.

The number-average molecular weights were determined from the carboxyl and amine end groups of the polyamides by anhydrous titration [7].

We utilized the diols containing siloxane linkages (II, VI) for the preparation of new types of potentially useful polyurethanes containing long-chain alkyl fatty acid residues in their backbone.

The polyurethanes were synthesized by heating the diols at 200 to 240°C with 2,4-toluene diisocyanate, 4,4'-diphenylmethane diisocyanate, and 1,6-hexane diisocyanate (Table 4).



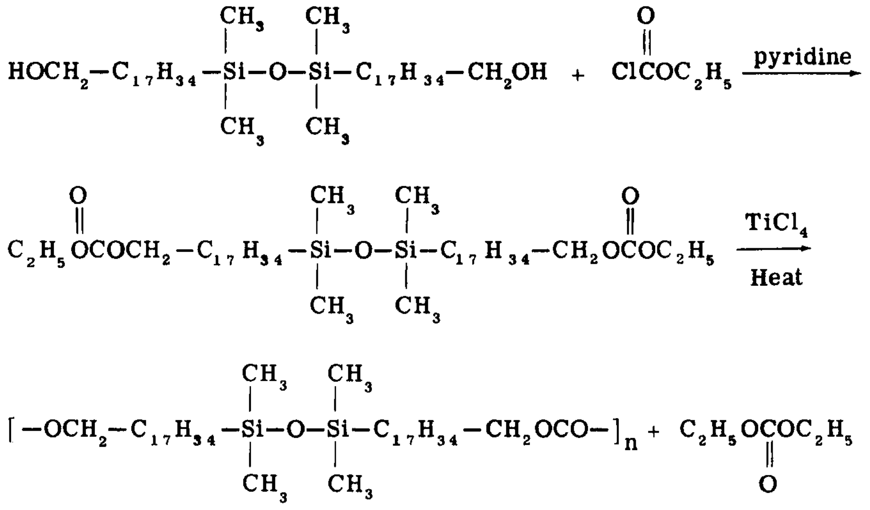
$$\mathrm{nHOCH_2-C_{17}H_{34}-\underset{CH_3}{\overset{CH_3}{Si}}-O-\underset{CH_3}{\overset{CH_3}{Si}}-C_{17}H_{34}-CH_2OH + nOCN-R-NCO} \rightarrow$$

$$\mathrm{[-OCH_2-C_{17}H_{34}-\underset{CH_3}{\overset{CH_3}{Si}}-O-\underset{CH_3}{\overset{CH_3}{Si}}-C_{17}H_{34}-CH_2-OCONH-R-NHCO-]_n}$$

The polyurethanes were soluble in methylene chloride and films were cast from solution.

Part of the polymers were thermally stable up to 300°C, as indicated by the date obtained from TGA (Fig. 1).

The polycarbonates (Table 5) were synthesized by reacting the diols (II, VI, IX) with ethylchloroformate in the presence of pyridine to give the mixed carbonate ester which on heating in vacuo in the presence of titanium tetrachloride [8] gave the polycarbonate:



$$\mathrm{HOCH_2-C_{17}H_{34}-\underset{CH_3}{\overset{CH_3}{Si}}-O-\underset{CH_3}{\overset{CH_3}{Si}}-C_{17}H_{34}-CH_2OH + Cl\overset{O}{\overset{\|}{C}}OC_2H_5} \xrightarrow{\mathrm{pyridine}}$$

$$\mathrm{C_2H_5O\overset{O}{\overset{\|}{C}}OCH_2-C_{17}H_{34}-\underset{CH_3}{\overset{CH_3}{Si}}-O-\underset{CH_3}{\overset{CH_3}{Si}}-C_{17}H_{34}-CH_2O\overset{O}{\overset{\|}{C}}OC_2H_5} \xrightarrow[\mathrm{Heat}]{\mathrm{TiCl_4}}$$

$$\mathrm{[-OCH_2-C_{17}H_{34}-\underset{CH_3}{\overset{CH_3}{Si}}-O-\underset{CH_3}{\overset{CH_3}{Si}}-C_{17}H_{34}-CH_2OCO-]_n + C_2H_5O\underset{O}{\underset{\|}{C}}OC_2H_5}$$

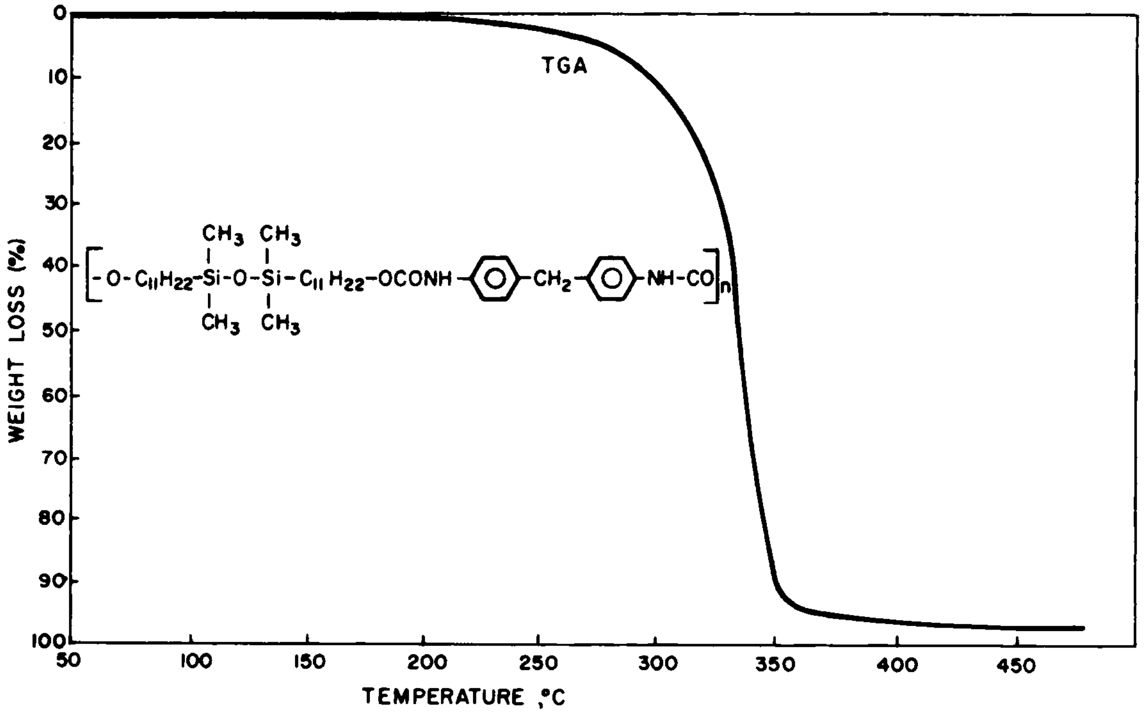


FIG. 1. TGA of polyurethane from 1,3-bis(11-hydroxyundecyl)tetramethyldisiloxane and 2,4-toluene diisocyanate.

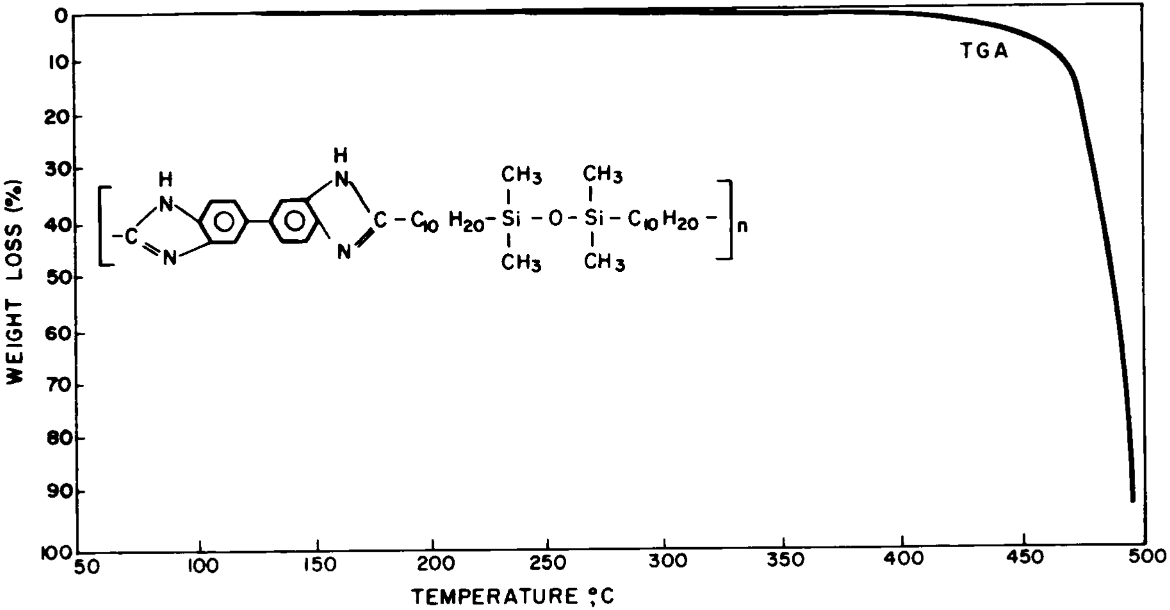
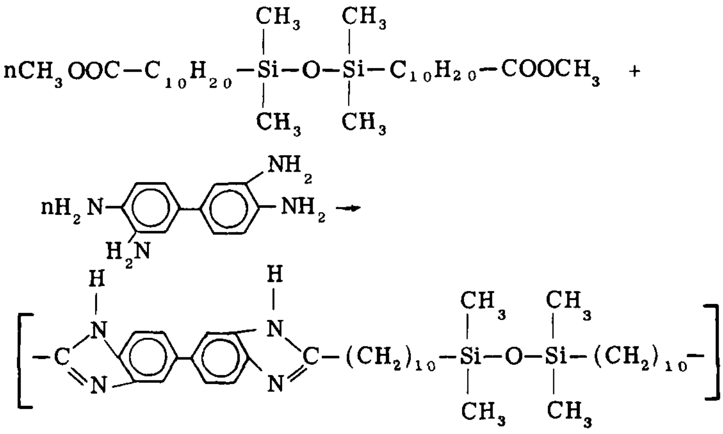


FIG. 2. TGA of polybenzimidazole from 1,3-bis(10-carbomethoxydecyl)tetramethyldisiloxane and 3,3'-diaminobenzidine.

The polycarbonates obtained were viscous materials which were soluble in benzene and chloroform.

Some polybenzimidazoles containing disiloxane linkages and fatty acid residues in their backbone were synthesized (Table 6) by heating the diesters at 200 to 250°C with 3,3'-diaminobenzidine:



Some of the polymers were thermally stable up to 300°C, as indicated by the data obtained from TGA (Fig. 2).

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