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Synthesis and characterization of octakis(3,3,3trifluoropropyldimethylsiloxy)cyclotetrasiloxane (**I**), octakis[tridecafluoro-2,2,5,5-tetramethyl-2,5disilatridecanoxy]cyclotetrasiloxane (**II**) and octakis(7-pentafluorophenyl-2,2,5,5-tetramethyl-2,5disilaheptanoxy)cyclotetrasiloxane (**III**)

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To Professor Chengye Yuan, distinguished member of the Chinese Academy of Science, a creative and highly productive organo-phosphorus chemist on the occasion of his 80th birthday.

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Abstract

Octakis(3,3,3-trifluoropropyldimethylsiloxy)cyclotetrasiloxane (**I**), octakis[tridecafluoro-2,2,5,5-tetramethyl-2,5-disilatridecanoxy]cyclotetrasiloxane (**II**), and octakis(7-pentafluorophenyl-2,2,5,5-tetramethyl-2,5-disilaheptanoxy)cyclotetrasiloxane (**III**) have been prepared and characterized. Reaction of octakis[chloro calcium oxy]cyclotetrasiloxane ($Ca_8Si_4O_{12}Cl_8$) with 3,3,3-trifluoroproyldimethylchlorosilane yields **I**, **II**, and **III**, on the other hand, have been prepared by platinum catalyzed hydrosilylation reactions of octakis(vinyldimethylsiloxy)cyclotetrasiloxane with tridecafluoro-1H,1H,2H,2H-octyldimethylsilane and 2-pentafluorophenyl-1H,1H,2H,2H-ethyldimethylsilane, respectively.

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1. Introduction

Despite numerous useful properties, including hydrophobicity, low glass transition temperature (T_g), low surface energy, and biocompatibility among others, polysiloxane elastomers are often mechanically weak and lack sufficient toughness for many practical applications. For these reasons, polysiloxanes are often reinforced with high surface area (~300 m²/g) inorganic materials, such as fumed silica, to form composite materials [1,2]. To optimize properties, the non-polar polysiloxane and the highly polar fumed silica must be made mutually compatible. This is often achieved by trimethylsilylation of fumed silica by treatment with hexamethyldisilazane or other similar trimethylsilylation

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reagents. This converts the hydrogen-bonding, polar surface silanol (Si–OH) groups of the fumed silica into non-polar trimethylsiloxy (O–Si(CH₃)₃) units [3–5].

Recently, there has been considerable interest in silsesquioxane materials as nano-scale molecular reinforcing materials for polymer composites [6]. Silsequioxanes have a cubic framework comprised of 8 silicon atoms and 12 oxygen atoms. Typically, the silicon atoms found at the corners of the cube have an organic group bonded to them. These organic groups may make the silsesquioxane compatible in blends with organic polymers or they may be used to covalently attach the silsesquioxane to the polymer backbone as a pendant group or as an integral part of the polymer backbone [7–11].

We have been interested in the preparation of cyclotetrasiloxanes in which each silicon atom of the cyclic core is substituted with two siloxy groups. For example, we have prepared octakis(vinyldimethylsiloxy)cyclotetrasiloxane by

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reaction of $Ca_8Si_4O_{12}Cl_8$, octakis[chloro calcium oxy]cyclotetrasiloxane, with an excess of vinyldimethylchlorosilane [12]. In this, each silicon atom of the core has a total of four oxygen atoms bonded to it and as such is a silicate silicon atom or a Q unit. In such systems, a total of eight siloxy groups are attached to the four core silicon atoms. In this sense, these materials are analogous to silsesquioxanes.

2. Results and discussion

In this paper, we report the preparation and characterization of octakis(3,3,3-trifluoropropyldimethylsiloxy)cyclotetrasiloxane (**I**), octakis[tridecafluoro-2,2,5,5-tetramethyl-2,5-disilatridecanoxy]cyclotetrasiloxane (**II**), and octakis(7pentafluorophenyl-2,2,5,5-tetramethyl-2,5-disilaheptanoxy)cyclotetrasiloxane (**III**).

I was prepared by the direct displacement reaction of $Ca_8Si_4O_{12}Cl_8$ with 3,3,3-trifluoropropyldimethylchlorosi-

lane. The key starting material $Ca_8Si_4O_{12}Cl_8$ is prepared by heating wollastonite, an inexpensive linear calcium silicate mineral, to 750–800 °C in the presence of a stoichiometric amount of calcium chloride dihydrate (CaCl₂·2H₂O) [13]. **II** and **III**, on the other hand, have been prepared by Pt-catalyzed hydrosilylation reactions between octakis-(vinyl-dimethylsiloxy)cyclotetrasiloxane, prepared by reaction of $Ca_8Si_4O_{12}Cl_8$ and vinyldimethylchlorosilane, and tridecafluoro-1H,1H,2H,2H-octyldimethylsilane or 2-pentafluorophenyl-1H,1H,2H,2H-ethyldimethylsilane, respectively (Fig. 1). These novel silicate cyclotetrasiloxane derivatives **I**, **II**, and **III** were fully characterized.

3. Experimental

¹H, ¹³C, ¹⁹F, and ²⁹Si NMR were obtained on a Bruker AMX-500 spectrometer. Five percent w/v C_6D_6 solutions were used to obtain ¹H and ¹⁹F NMR spectra. Fifty percent



Fig. 1. Synthesis of I and III.

w/v C₆D₆ solutions were used to obtain ¹³C and ²⁹Si NMR spectra. ¹³C NMR spectra were obtained with broad band proton decoupling. ²⁹Si NMR were acquired using a NONOE pulse sequence with a 60 s delay. ¹H and ¹³C NMR spectra were internally referenced to residual C₆D₆. ²⁹Si NMR spectra were externally referenced to TMS. ¹⁹F NMR spectra were externally referenced to CFCl₃. IR spectra of neat films of liquids on AgCl plates or KBr pellets of solids were recorded on a Perkin–Elmer spectrum 2000 FT–IR spectrometer.

High-resolution chemical ionization mass spectra were run at the University of California at Riverside Mass Spectroscopy Facility on a MALDI DE-STR high-resolution instrument. Ionization was achieved by attachment of sodium cations. Exact masses were determined by peak matching against known masses of perfluorokerosene.

Gel permeation chromatography analysis of the molecular weight distribution (M_w/M_n) of **II** and **III** were performed on a Waters system comprised of a Waters 510 HPLC pump, an 820 Maxima control system, and a R401 differential refractometer. The eluting solvent was toluene at a flow rate of 0.6 mL/min. Two 7.8 mm × 300 mm Styragel HT 6E and HMW 6E column in series were used for the analysis. The retention times were calibrated against known mono-dispersed polystyrene standards 891 000, 212 400, 29 300, 3 680, 770 g/mol.

Glass transition temperatures (T_g) and crystalline melting temperatures (T_m) of the cyclic silicate oligomers were determined on a Shimadzu DSC-50. The differential scanning calorimeter (DSC) was calibrated from the heat of transition $(-87.06 \,^{\circ}\text{C})$ and T_m (6.54 $\,^{\circ}\text{C})$ of cyclohexane [14], as well as the T_g $(-125 \,^{\circ}\text{C})$ of polydimethylsiloxane (PDMS) [15]. After equilibration at $-150 \,^{\circ}\text{C}$ for 15 min, the temperature was increased at a rate of 10 $\,^{\circ}\text{C/min}$ from -150 to 25 $\,^{\circ}\text{C}$.

Wollastonite, vinyldimethylchlorosilane, 3,3,3-trifluoropropyldimethylchlorosilane, and tridecafluoro-1H,1H,2H,2Hoctyldimethylchlorosilane were purchased from Gelest. Pentafluorostyrene was obtained from SynQuest Labs, Inc. Karstedt catalyst, a 1,3-divinyltetramethyldisiloxane complex of platinum (2% Pt) in xylene, was acquired from United Chemical Technologies, Inc.

All reactions were run in flame dried flasks equipped with a Teflon covered magnetic stir bar under nitrogen.

3.1. Octakis[chloro calcium oxy]cyclotetrasilicate [Ca₈Si₄O₁₂Cl₈]

 $Ca_8Si_4O_{12}Cl_8$ was prepared by heating wollastonite and a stoichiometric amount of $CaCl_2 \cdot 2H_2O$ in a carbon boat which was placed in a quartz tube in a Hoskins electric tube furnace. The tube and its contents were heated for 17 h at 750–800 °C under a continuous flow of nitrogen [13].

3.2. Octakis(vinyldimethylsiloxy)cyclotetrasiloxane [12]

Octakis (vinyl dimethyl siloxy) cyclotetrasiloxane was pre $pared by reaction of Ca_8Si_4O_{12}Cl_8 \ (16.25 \ g, \ 17.2 \ mmol)$ with vinyldimethylchlorosilane (4.15 g, 34 mmol) in 100 mL of dry acetone. The mixture was refluxed for 24 h. The solution was then filtered and the volatiles removed by evaporation under reduced pressure. The residue was purified by flash column chromatography on silica gel with hexane as the eluting solvent. In this way, a 24% yield, 1.03 g of white crystals, mp 160 °C were obtained. ¹H NMR δ : 0.18 (s, 6H), 5.75 (dd, 1H, J = 20 and 4 Hz), 5.92 (dd, 1H, J = 15 and 4 Hz), 6.13 (dd, 1H, J = 20 and 15 Hz). ¹³C NMR δ : 0.24, 132.1, 138.9. ²⁹Si NMR δ : -1.52 (s, 2Si), -108.6 (s, 1Si). IR n: 3185, 3054, 3015, 2961, 1913, 1597, 1065, 771 cm⁻¹. Chemical ionization HRMS calculated for [C₃₂H₇₂O₁₂Si₁₂Na]⁺: 1007.21474. Found: 1007.21820.

3.3. Tridecafluoro-1H,1H,2H,2H-octyldimethylsilane [16]

Diethyl ether (15 mL) and LiAlH₄ (0.17 g, 4.7 mmol) were placed in a 50-mL three neck round bottom flask equipped with a pressure equalizing addition funnel and a reflux condenser. A solution of diethyl ether (10 mL) and tridecafluoro-1H,1H,2H,2H-octyldimethylchlorosilane (4.5 g, 9.4 mmol) was placed in the addition funnel. The solution was added dropwise over 30 min. The reaction was stirred at room temperature for 1 h. The reaction mixture was then filtered and the diethyl ether removed by distillation. The residue was then distilled through a 5-cm vacuum jacketed Vigreux column under reduced pressure. A fraction bp 55 °C/20 mm, 2.6 g, 68% yield was obtained. ¹H NMR δ : 0.135 (d, 6H, J = 3.7 Hz), 0.834 (ddd, 2H, J = 9.0, 8.0 and 3.5 Hz), 2.07 (m, 2H), 3.92 (septet, 1H, J = 3.5 Hz). ¹³C NMR δ : -4.82, 3.76, 26.44 (t, $J_{C-F} = 2.36$ Hz), 106.5 (m), 109.0 (m), 111.4 (m), 116.2 (m), 118.4 (m), 120.4 (m). ¹⁹F NMR δ : -81.36 (t, 3F J = 11 Hz), -116.5 (pentad, 2F, J = 14 Hz), -122.5 (br.s, 2F), -123.4 (br.s, 2F), -123.9 (br.s, 2F), -126.7 (m, 2F). ²⁹Si NMR δ: -11.3 (s). IR n: 2964, 2911, 2125(Si-H), 1443, 1425, 1238, 1194, 1145, 917, 891 cm^{-1} .

3.4. (Pentafluorophenyl)ethyldimethylchlorosilane [17]

Pentafluorostyrene (5 g, 25 mmol), dimethylchlorosilane (3 g, 31 mmol), and Karstedt's catalyst (5 mL) were placed in a 25-mL round bottom flask equipped with a reflux condenser. The reaction was stirred at reflux for 3 h. Excess dimethylchlorosilane was removed by evaporation under reduced pressure. The residue was distilled through a 5-cm vacuum jacketed Vigreux column under reduced pressure. A fraction bp 56 °C/0.75 mm, 6.2 g, 86% yield was obtained. ¹H NMR δ : 0.47 (s, 6H), 1.14 (m, 2H), 2.81 (t, 2H, J = 8.5 Hz). ¹³C NMR δ : 1.36, 16.19, 116.9 (tt, $J_{C-F} = 19$ and 2 Hz), 137.25 (d, $J_{C-F} = 23$ and 10 Hz), 139.63 (d, $J_{C-F} = 250$ Hz), 144.85 (d, $J_{C-F} = 244$ Hz). ¹⁹F NMR δ : -145.5 (dd, 2F, J = 23 and 10 Hz), -158.6 (t, 1F, J = 20 Hz), -163.3 (dt, 2F, J = 26 and 9 Hz). ²⁹Si NMR δ : 30.53.

3.5. (Pentafluorophenyl)ethyldimethylsilane

Diethyl ether (15 mL) and LiAlH₄ (0.22 g, 5.5 mmol) were placed in a 50-mL three neck round bottom flask equipped with a reflux condenser and a pressure equalizing addition funnel. A solution of 2-(pentafluorophenyl)ethyldimethylchlorosilane (6.2 g, 21 mmol) and diethyl ether (10 mL) was placed in the addition funnel and added dropwise over 30 min. The reaction was allowed to stir for 1 h at room temperature. The solution was then filtered and the diethyl ether was removed by evaporation under reduced pressure. The residue was then distilled through a 5-cm vacuum jacketed Vigreux column under reduced pressure. A fraction bp 30 °C/0.5 mm, 4.1 g, 75% yield was obtained. ¹H NMR δ : 0.128 (d, 6H, J = 4 Hz), 0.91 (m, 2H), 2.73 (tt, 2H, J = 8.7 and 1.2 Hz), 3.88 (septet, 1H, J = 3.7 Hz). ¹³C NMR δ : -4.79, 14.88, 17.48, 117.79 (tq, $J_{C-F} = 18$ and 4 Hz), 136.56 (d, $J_{C-F} = 242$ Hz), 139.52 (d, $J_{C-F} = 250 \text{ Hz}$), 144.89 (d, $J_{C-F} = 242 \text{ Hz}$). ¹⁹F NMR δ : -145.81 (dd, 2F, J = 20 and 10 Hz), -159.3 (t, 1F, J = 21 Hz), -163.69 (dt, 2F, J = 23 and 9 Hz). ²⁹Si NMR δ: -12.92. IR n: 2962, 2899, 2120 (Si-H), 1656, 1520, 1505, 1253, 1155, 1120, 988, 955, 915, 883, 839, 605 cm^{-1} .

3.6. Octakis(3,3,3-trifluoropropyldimethylsiloxy)cyclotetrasiloxane

Ca₈Si₄O₁₂Cl₈ (5 g, 5.3 mmol) was placed in a 500 mL round bottom flask equipped with a reflux condenser. 3,3,3-Trifluoropropyldimethylchlorosilane (10 g, 55 mmol) and dry acetone (150 mL) were added. The mixture was refluxed for three days. The solution was then filtered and the volatiles were removed by evaporation under reduced pressure. The residue was passed through a silica gel column with hexane/dichloromethane (12:1). The volatiles were removed by evaporation under reduced pressure to yield 0.5 g, 6% yield of a white solid, mp 294 °C. ¹H NMR δ : 0.185 (s, 48H), 0.825 (m, 16H), 2.05 (m, 16H). ¹³C NMR δ: -0.49 (s), 9.75 (s), 28.25 (q, $J_{C-F} = 30$ Hz), 127.76 (q, J_{C-F} = 275 Hz). ¹⁹F NMR δ : -69.27 (t, $J_{\text{H-F}}$ = 10.8 Hz). ²⁹Si NMR δ: 11.4 (s, 2 Si), -108.6 (s, 1Si). IR n: 2964, 2907, 1446, 1368, 1315, 1263, 1207, 1121, 1066, 902, 843, 791 cm⁻¹. HRMS calculated for $[C_{40}H_{80}O_{12}F_{24}NaSi_{12}]^+$: 1,567.2390. Found: 1,567.2372.

In a separate experiment, $Ca_8Si_4O_{12}Cl_8$ (5 g, 5.3 mmol), 3,3,3-trifluoropropyldimethylchlorosilane (10 g, 55 mmol) and dry acetone (150 mL) were added into a 250 mL rb flask fitted with a reflux condenser and a Tekmar sonic disruptor micro-tip working at a 20 kHz frequency. Ultra sonic waves were passed into the solution in 5 s pulses of 1 s each. The solution was stirred for 2 h. The salts were filtered through a sintered glass funnel and washed three times with pentane. The combined organic solution was fractionally distilled through a 10 cm vacuum jacketed Vigreux column. Excess 3,3,3-trifluoropropyldimethylchlorosilane, bp 118 °C, and bis(3,3,3-trifluoropropyl)tetramethyldisiloxane [18], bp 51 °C/4 mm, were recovered. The residue, 0.4 g, was passed through a silica gel column with dichloromethane to yield **I** whose spectral properties agreed with those reported above.

3.7. Octakis[tridecafluoro-2,2,5,5-tetramethyl-2,5-disilatridecanoxy]cyclotetrasiloxane

Octakis[vinyldimethylsiloxy]cyclotetrasiloxane (0.5 g. 0.5 mmol) and tridecafluoro-1H,1H,2H,2H-octyldimethylsilane (2.05 g, 5 mmol) were placed in a 25 mL round bottom flask sealed with a rubber septum. Karstedt's catalyst $(5 \ \mu L)$ was added to the solution. The reaction was stirred for 2 h or until the Si-H signal at \sim 2100 cm⁻¹ in the IR had disappeared. The mixture was then diluted with 10 mL of dichloromethane. The solution was purified by flash chromatography on a short silica gel column to remove the Pt catalyst. The volatiles were removed by evaporation under reduced pressure. In this way, a clear liquid, 1.8 g, 85% yield $M_{\rm w}/M_{\rm n} = 3500/3300$ and $T_{\rm g} = -67$ °C was obtained. ¹H NMR 5: 0.011 (s, 6H), 0.112 (s, 6H), 0.449 (s, 4H), 0.69-0.79 (m, 2H), 1.87–2.08 (m, 2H). ¹³C NMR δ: -4.46, -0.79, 3.99, 6.28, 9.80, 25.98 (t, J = 24 Hz), 108.9 (m), 111.2 (m), 113.5 (m), 116.4 (m), 118.6 (t, J = 3.2 Hz), 120.72 (q, J =31 Hz). ¹⁹F NMR δ : -81.64 (t, 3F, J = 8 Hz), -116.8 (m, 2F), -122.65 (br.s, 2F), -123.6 (br.s, 2F), -123.9 (br.s, 2F), -126.9 (br.s, 2F). ²⁹Si NMR δ: 10.86 (s, 2Si), 5.52 (s, 2Si), -108.0 (s, 1Si). IR n: 2959, 2909, 1351, 1240, 1120, 1069, 899, 837 cm⁻¹. HRMS calculated for $[C_{112}H_{160}F_{104}O_{96}-$ SiNa]⁺: 4259. Found: 4259.

3.8. Octakis(7-pentafluorophenyl-2,2,5,5-tetramethyl-2,5disilaheptanoxy)cyclotetrasiloxane

Octakis[vinyldimethylsiloxy]cyclotetrasiloxane (0.5 g, 0.5 mmol) and 2-(pentafluorophenyl)ethyldimethylsilane (2.05 g, 5 mmol) were placed in a 25 mL round bottom flask equipped with a rubber septum. Karstedt's catalyst $(5 \,\mu L)$ was added. The reaction was stirred for 2 h or until the Si–H signal in the IR at $\sim 2100 \text{ cm}^{-1}$ had disappeared. The mixture was then diluted with 10 mL of dichloromethane. The solution was purified by flash chromatography on a short silica gel column to remove the Pt catalyst. The volatiles were removed by evaporation under reduced pressure. In this way, 1.1 g, 66% yield, $M_w/M_n =$ 2300/2200 and $T_g = -63 \,^{\circ}\text{C}$ was obtained. ¹H NMR δ : 0.011 (s, 6H), 0.11 (s, 6H), 0.46 (s, 4H), 0.81 (m, 2H), 2.64 (m, 2H). 13 C NMR δ : -3.81, -0.28, 6.79, 10.33, 1606, 17.46, 118.86 (t, $J_{C-F} = 27$ Hz), 137.38 (d, $J_{C-F} = 232$ Hz), 139.38 (d, $J_{C-F} = 237$ Hz), 144.68 (d, $J_{C-F} = 242$ Hz). ¹⁹F NMR δ : -146.17 (dd, 2F, J = 214 and 9 Hz), -159.58 (t, 1F, J = 20 Hz), -163.78 (dt, 2F, J = 23 and 9 Hz). ²⁹Si NMR δ: 10.82 (s, 2Si), 4.69 (s, 2Si), -108.17 (s, 1Si). IR n: 2958, 2909, 1654, 1505, 1408, 1251, 1054, 986, 901, 830 cm^{-1} .

4. Conclusions

Efficient synthetic routes to prepare octakis(3,3,3-trifluoropropyldimethylsiloxy)cyclotetrasiloxane (I), octakis[tridecafluoro-2,2,5,5-tetramethyl-2,5-disilatridecanoxy]cyclotetrasiloxane (II), and octakis(7-pentafluorophenyl-2,2,5,5tetramethyl-2,5-disilaheptanoxy)cyclotetrasiloxane (III) are reported. The direct displacement reaction of Ca₈Si₄O₁₂Cl₈ with 3,3,3-trifluoropropyldimethylchlorosilane gave I in a low yield (6%). An attempt to increase the yield and shorten the reaction time by applying ultra sonic waves to the reaction media had little effect. Ultrasound has been shown to improve yields and accelerate many heterogeneous reactions [19]. The yield of the reaction did not improve and the reasons for this are not fully understood. On the other hand, II and III have been prepared in higher overall yields by a two-step process. The first step involves the synthesis of octakis(vinyldimethylsiloxy)cyclotetrasiloxane by the direct displacement reaction (24% yield) of Ca₈Si₄O₁₂Cl₈ with vinyldimethylchlorosilane. II and III are then produced by the platinum catalyzed hydrosilylation reaction between octakis(vinyldimethylsiloxy)cyclotetrasiloxane and tridecafluoro-1H,1H,2H,2H-octyldimethylsilane in 85% yield and 2-(pentafluorophenyl)ethyldimethylsilane in 66% yield, respectively. This work makes these novel organofluorine substituted cyclic silicate materials available for further study.

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