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Synthesis of oligosiloxane Q_2M_6 [$Q = (SiO_{4/2})_4$, $M = Me_3SiO_{2/3}$] from trimethylsilylation of complex silicates

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Because natural silicates are potential sources of oligosiloxanes of the type QxMy, a collection of natural silicates of similar structure was used to obtain mainly Q_2M_6 oligosiloxanes by trimethylsilylation. The tailings from silver and gold flotation beneficiation provide an important silicate source. The main silicate phase reactive to trimethylsilylation was found to be anorthite. Other minerals containing silicates with a structure similar to anorthite (labradorite and augite) were also used as a Q_2M_6 source. This paper presents a full characterization of Q_2M_6 as well as information related to the residual solids from the TMS reactions. Copyright © 2006 John Wiley & Sons, Ltd.

KEYWORDS: oligosiloxanes; Q_2M_6; natural silicates; TMS reactions

INTRODUCTION

Silicon, as SiO_2 and silicates, is the second most abundant element in the earth's crust. The production of polysiloxanes and almost all organosilanes comes from elemental silicon. The transformation of SiO_2 to silicon in the well-known carbothermic process requires huge amounts of energy with temperatures of around 1700 °C.[1] The direct process (silicon reacting with methyl chloride in the presence of a copper catalyst) is the most useful method in industry for obtaining polysiloxane monomers.[2] The methylchlorosilanes are the most important of these monomers with dimethyldichlorosilane [$(CH_3)_2SiCl_2$] the most important of them. The hydrolysis of this monomer generates polymethylsiloxanes, one of the most important classes of industrial materials.[3] The high energy demand of this process encourages investigation of low-energy alternative routes.

The trimethylsilylation (TMS) of natural silicates has been considered as an alternate way to incorporate organic groups into the silicate structure, in particular the incorporation of [$(CH_3)_3SiO$-] groups using the method described by Lenz[4] in 1964. This method was developed to determine the silicate structure in which the silicate source reacts in an acidic medium (HCl–water–isopropyl alcohol) with a silylation reagent (mainly hexamethyldisiloxane or trimethylchlorosilane) to form stable trimethylsilylated species of the type QxMy [$Q = SiO_{4/2}$ and $M = (CH_3)_3SiO_{1/2}$]. The product obtained maintains in sufficient percentages the structure of the starting structure of the silicate.[5–7] Figure 1 illustrates the TMS reaction.

The industrial importance of polymethylsiloxanes in industry and their applications in daily life[8] challenge scientists to look for economical starting materials. Our research group has been looking for applications involving modification of tailings from the silver and gold sulfide ores in central Mexico. The stored tailings are considered pollutants because of their heavy-metal content as well as the high concentration of finely divided quartz and silicates. Recently, we reported the potential use of these tailings as a source of QxMy oligosiloxanes.[9] We mention in that report preliminary evidence of the possibility of obtaining the oligosiloxane Q_2M_6 from reactions of TMS with anorthite. Because anorthite is known to be highly soluble in HCl, it is an excellent candidate for silylation with TMS. Thus, we report here further details of this reaction. Product characterizations were carried out using NMR (^{29}Si, ^{1}H and ^{13}C in solution), IR, GC and GC/MS.

The residual solids from TMS reactions were characterized using several techniques, namely, atomic absorption, XRD, solid-state ^{29}Si NMR (MAS and CPMAS), thermal analysis

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(DTA, TGA and DGT), IR (DRIFTS), textural properties and contact angle measurements.

Q$_2$M$_6$ synthesis from TMS reaction with anorthite present in the tailings is supported by the TMS of other natural silicates with structures similar to anorthite ($CaAl_2Si_2O_8$). The silicates selected were labradorite [$(Na,Ca)(Al,Si)_4O_8$] (also known as calcareous anorthite) and a complex silicate with augite [$Ca(Mg,Fe)Si_2O_6$] (a pyroxene) as the main component (see Fig. 2).

EXPERIMENTAL

Authentic samples of QM$_4$ and Q$_2$M$_6$ were obtained from natural samples of the orthosilicate olivine, which is one of the most reactive minerals toward TMS. The olivine samples were obtained from Durango in Northern Mexico. They proved to be quite useful for characterizing the oligosiloxanes and the other silicates from tailings.

The olivine was milled in an alumina ball mill to an average particle size of 74 µm. In the TMS reaction, a mixture of 250 ml HCl (36%, density = 1.18), 500 ml isopropyl alcohol and 333 ml hexamethyldisiloxane was stirred for 1 h at room temperature. Next 50 g of olivine in 200 ml of water were added to the mixture and stirred at reflux (68 °C) for 6 h. The final mixture was left to settle and then filtered. The organic phase was extracted with $CHCl_3$ twice to separate the products into two phases. The organic phase was distilled at atmospheric pressure (102 °C) to recover the excess hexamethyldisiloxane. The residue contained the oligosiloxane Q$_x$M$_y$ mixture. QM$_4$ was purified by distillation (85–100 °C) and Q$_2$M$_6$ purified by sublimation (140 °C, 12 Torr).

The silicate samples used in this study were from the tailings of a local mining company in Guanajuato Mexico. The labradorite was a donation from the School of Mines, University of Guanajuato, Guanajuato, Mexico and the silicate-containing augite was donated by the Centro de Investigaciones en Química Inorgánica, University of Guanajuato, Guanajuato, Mexico. The latest sample was obtained from ores located in the region of Saltillo State, Northern Mexico.

To obtain Q$_2$M$_6$ as the principal product, in a typical TMS reaction 4 g silicate samples were stirred in 16 ml of water for 1 h, and then added to a mixture of 40 ml isopropyl alcohol, 20 ml concentrated HCl (density = 1.18) and 26.6 ml hexamethyldisiloxane. This mixture was stirred continuously for 1 h at room temperature. The reaction mixture was then

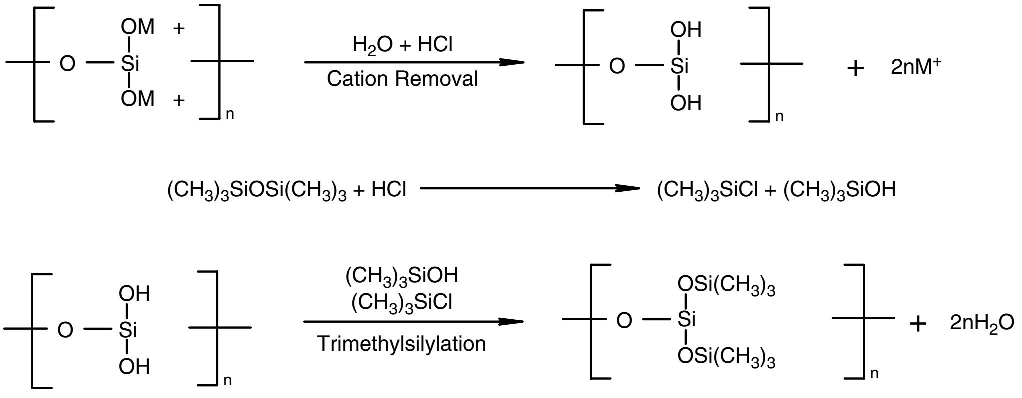
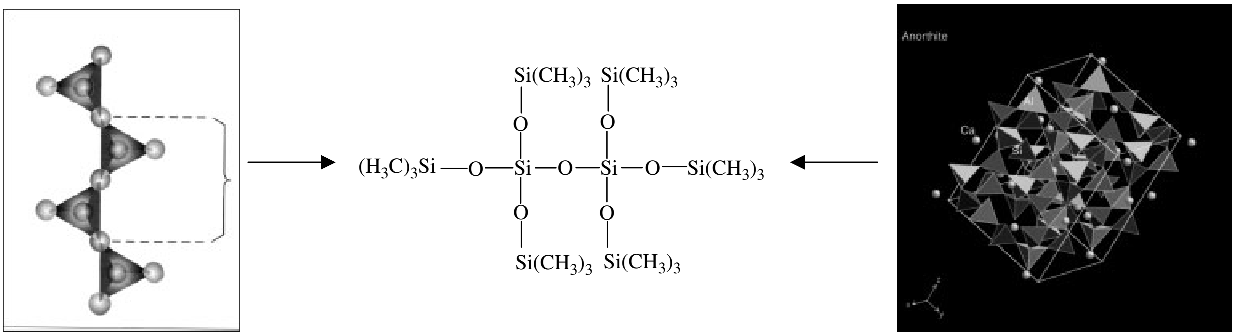


Figure 1. The trimethylsilylation reaction (TMS).



Augite structure

Q$_2$M$_6$

Anorthite structure

Figure 2. Natural silicates sources for Q$_2$M$_6$.

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stirred at reflux (68 °C). The stirring time was related to the type of silicate sample. The crude reaction products were cooled to room temperature, filtered and left to settle until separation of three phases occurred: an organic layer, the aqueous phase, and a layer containing suspended residual silicate. The residual silicate was washed twice with distilled water and twice with isopropyl alcohol before drying in an oven for 12 h. Then the residual solid was characterized using atomic absorption, solid-state NMR (^{29}Si and ^{27}Al) and DRX. Table 1 illustrates representative TMS reactions and the conditions used.

The oligosiloxanes present in the organic phase were extracted with chloroform. The solvents were distilled off at reduced pressure (40–50 °C, 12 mmHg) and the residue from the distillation characterized by GC, GC/MS, IR and NMR (^{29}Si, ^{13}C and ^{1}H). The cations removed from the silicate samples were monitored by atomic absorption analysis of the aqueous phase.

Analytical techniques

Gas chromatography and mass spectroscopy–gas chromatography GC data were obtained using an Agilent system 6890 GC with a FID detector. The MS-GC data were collected using a Varian CP-3800 coupled to a MS detector Varian Saturn 2000. The detector was ionization by electronic impact.

Nuclear magnetic resonance spectrometry

The NMR spectra in $CDCl_3$ were obtained on a Varian Unity Plus multinuclear spectrometer at 300 MHz. For ^{29}Si spectra the sequence used was INEPT operating at 59.58 Hz, using an acquisition time of 2.0, a delay time of 5.0 and 7000 transients. The ^{13}C NMR spectra were collected at 75.430 Hz, acquisition time 1.815, delay time 4.0 and 1024 transients.

Infrared spectrometry

Solution IR spectra were recorded on a Perkin-Elmer 1600 FTIR in chloroform.

Atomic absorption spectrometry

Atomic absorption studies were performed in a TJA Solutions Solar Series Atomic Absorption spectrometer using the standard addition method. All samples were analyzed for Si, Al, Fe, K, Na, Mg and Ca.

Powder X-ray diffraction

X-ray powder diffraction studies were performed using an INEL, diffractometer Equinox model at 30 kV and 20 mA and also a Siemens D-5000 diffractometer at 35 kV and 20 mA. The scan rate was 2° 2θ/min, and a range of 5–80° 2θ.

Solid-state nuclear magnetic resonance

^{29}Si (MAS and CPMAS) spectra were obtained on a Varian Unity-plus 300 MHz NMR spectrometer operating at 59.58 MHz. A 7 mm diameter silicon nitride rotor with kel-F caps was used. The rotor spin rate was 4 kHz, with a delay time of 6 s and 5000 transients. Talc was used as an external reference (−99 ppm). The NMR spectra were recorded before and after the TMS reaction.

For ^{29}Si CPMAS NMR, the spectra were recorded at 59.58 Hz, acquisition time 0.05 s, delay time 4.0, 8000 transients, and contact time 2500. The NMR spectra were obtained before and after the TMS reaction.

Infrared spectrometry of solid samples

IR spectra of solid samples were recorded using a Perkin Elmer 1600 FTIR spectrometer. The detector employed was deuterated triglycine (DTGS) using the diffuse reflectance (DRIFTS) accessory.

Table 1. TMS Reaction conditions

Experiment	Silicate	Initial weight (g)	Final weight (g)	Yield (%)	HMDS (ml)	HCl (ml)	Water (ml)	2-Propanol (ml)	Reaction time (h)	Transference reagent	Particle size (mesh)
1	Labradorite	0.4589	0.4546	0.93	8	12	4	40	4	2-Propanol	−180
2		0.4997	0.4597	8.00	8	12	4	40	24	2-Propanol	−180
3		0.4822	0.3965	17.77	8	12	4	40	48	2-Propanol	−180
4		0.4272	0.3431	19.69	8	12	4	40	72	2-Propanol	−180
5	Augite	5.0045	3.8953	22.16	33.25	25	20	50	6	2-Propanol	−180
6		5.0207	3.8375	23.57	33.25	25	20	50	12	2-Propanol	−180
7		5.0378	3.5663	29.21	33.25	25	20	50	24	2-Propanol	−180
8		5.0950	3.7364	26.67	33.25	25	20	50	6	2-Propanol	−180
9		5.0114	3.6262	27.64	33.25	25	20	50	6	2-Propanol	−180
10		5.2028	4.0875	21.44	33.25	25	20	50	6	etanol	−180
11		3.3618	2.3199	30.99	33.25	25	20	50	6	2-Propanol	−300
12		5.0008	3.4704	30.60	33.25	25	20	50	6	2-Propanol	−325
13	Tailings	8.1145	7.6082	6.2	53.2	40	32	80	72	2-Propanol	−180
14		8.3159	7.1233	14.34	53.2	40	32	80	96	2-Propanol	−180

Thermal analysis studies were performed in a SDT system, model 2960 TA. Data were obtained under an air atmosphere (100 ml/min) with a heating rate of 10 °C/min. The standard used was α-alumina.

RESULTS AND DISCUSSION

The silicate samples were characterized by XRD and mineralogical methods before reaction with TMS. The mineralogical composition was calculated using the rational method.[10] The results are presented in Tables 2, 3 and 4. The main phases detected in the tailings were quartz, albite, orthoclase, chlorite and anorthite. Anorthite appears to be the most reactive phase for producing oligosiloxane Q_2M_6 as the main product. The augite sample was composed mainly of pyroxene, phyllosilicate, iron oxide, calcium carbonate and tectosilicates. The silicate labradorite was analyzed mainly by XRD. The main phase was calcic anorthite ($CaAl_2Si_2O_8$). The TMS reaction yield was 14% for tailings (anorthite), 20% for labradorite and 30% for augite. The reaction yield was calculated based on the weight of residual silicate after reaction with TMS.

Characterization of authentic simples of QM_4 and Q_2M_6

As described before, the TMS of olivine was used to obtain authentic samples of QM_4 and Q_2M_6, which were

Table 2. Mineralogical composition of tailings

Silicate	Mineral	Subclase	Formula	Weight (%)
Tailings	Quartz	Tectosilicate	SiO_2	58.32
	Albite	Tectosilicate	$NaAlSi_3O_8$	10.17
	Anorthite	Tectosilicate	$CaAl_2Si_2O_8$	2.97
	Orthoclase	Tectosilicate	$KAlSi_3O_8$	10.70
	Chlorite	Phylosilicate	$Mg_5Al(Si_3Al)O_{10}(OH)_8$	4.43
	Caolinite	Phylosilicate	$Al_2Si_2O_5(OH)_4$	1.01
	Calcite	Carbonate	$CaCO_3$	7.12
	Dolomite	Carbonate	$CaMg(CO_3)_2$	1.62
	Pirite	Sílfide	FeS_2	1.85
	hematite	Oxide	Fe_2O_3	0.47
	Ilmenite	Oxide	$FeTiO_3$	0.63
	Residual iron from milling balls	Oxide	Fe_2O_3	0.39
	Total			99.68

Table 3. Mineralogical composition of labradorite

Silicate	Mineral	Subclase	Formula	Weight (%)
Labradorite	Albite	Tectosilicate	$NaAlSi_3O_8$	41.69
	Anorthite	Tectosilicate	$CaAl_2Si_2O_8$	51.49
	Quartz	Tectosilicate	SiO_2	5.42
	Total			98.60

Table 4. Mineralogical composition of augite

Silicate	Mineral	Subclase	Formula	Weight (%)
Augite	Augite	Pyroxene	$Ca(Mg,Fe)Si_2O_6$	44.12
	Albite	Tectosilicate	$NaAlSi_3O_8$	16.78
	Orthoclase	Tectosilicate	$KAlSi_3O_8$	22.33
	Hematite	Oxide	Fe_2O_3	4.73
	Quartz	Tectosilicate	SiO_2	3.85
	Magnesite	Carbonate	$MgCO_3$	2.40
	Calcite	Carbonate	$CaCO_3$	0.39
	Chlorite	Phylosilicate	$Mg_5Al(Si_3Al)O_{10}(OH)_8$	1.88
	Total			99.48

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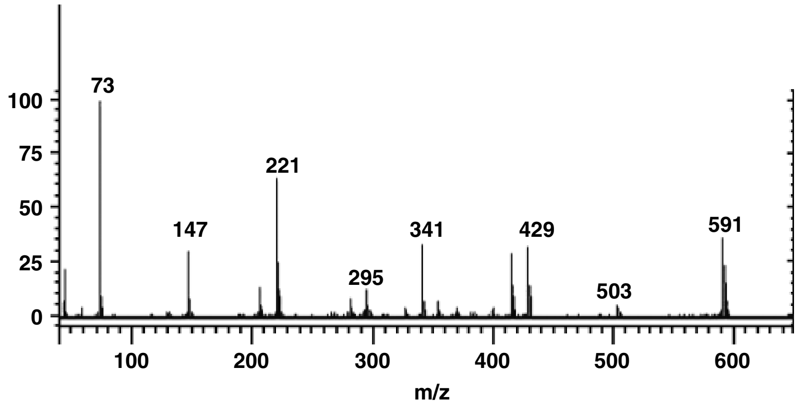

Figure 3. Mass spectra of Q_2M_6 from TMS reaction of olivine.

used as standards. GC chromatogram exhibits two main signals at retention times 8 and 13 min. These retention times are assigned to QM_4 and Q_2M_6. Q_2M_6 was isolated by sublimation (granular solid, m.p. 60 °C and b.p. 303 °C, MW 606). GC of the pure sample showed a retention time of 13 min. GC-MS was also used to characterize the authentic sample (Fig. 3). From the molecular ion $(CH_3)_{18}Si_8O_7$, the fragmentation pattern shows as main fragments (z/e): $[(CH_3)_{17}Si_8O_7]^+$ (591), $[(CH_3)_{14}Si_7HO_6]^+$ (503), $[(CH_3)_{11}Si_6H_2O_6]^+$, (429), $[(CH_3)_8Si_5H_3O_5]^+$ (341), $[(CH_3)_6Si_5H_2O_4]^+$ (296), $[(CH_3)_3HSi_4O_4]^+$ (222), $[(CH_3)_5Si_2O]^+$ (147) and $[(CH_3)_3Si]^+$ (73).

^{29}Si NMR in solution was a key analytical tool in the characterization. Signals were observed at −106 and 8 ppm assigned to Q and M groups respectively.[11] Solution FTIR spectroscopy shows characteristic bands for CH_3 (2959 cm^{-1}), $SiCH_3$ (1252 cm^{-1}), SiOSi (1073 cm^{-1}) and $Si(CH_3)_3$ (756 cm^{-1}).

Q_2M_6 from different silicates

This section presents a detailed discussion of silicate samples treated with the TMS reaction.

Tailings (anorthite)

A mineralogical analysis of tailings showed that they are composed mainly of quartz (around 60%) and aluminosilicates and silicates (30%), which means the phases reactive to TMS are quite limited. Nevertheless, the reactive phase was determined to be the anorthite $CaAl_2Si_2O_8$. The anorthite is appreciably soluble in HCl, and Q_2M_6 is the main product obtained. In order to verify the synthesis, a combination of GC, GC-MS and ^{29}Si NMR analysis was used: using GC, signals at 8 and 13 min retention times were observed as the main products in good accord with the authentic samples obtained from olivine. Of these, Q_2M_6 is the most abundant. For Q_2M_6 analysis done using GC-MS, the same fragmentation pattern was obtained: $[(CH_3)_{17}Si_8O_7]^+$ (591), $[(CH_3)_{14}HSi_7O_6]^+$ (503), $[(CH_3)_{12}HSi_6O_5]^+$ (429), $[(CH_3)_9H_2Si_5O_4]^+$ (341), $[(CH_3)_6H_2Si_5O_4]^+$ (296), $[(CH_3)_3HSi_4O_4]^+$ (222), $[(CH_3)_5Si_2O]^+$ (147) and $[(CH_3)_3Si]^+$ (73) (see Figs 4 and 5). Other signals detected by GC and GC-MS close to QM_4 and Q_2M_6 are partially trimethylsilylated derivatives. They were assigned as $Si_2O_7(SiMe_3)_2(Pr^i)_4$ and $Si_2O_7(SiMe_3)_4(Pr^i)_2$.[12] By the use of ^{29}Si NMR in solution, signals at 8 and −106 correspond to Q_2M_6 and the signals at 4 and −104 ppm to QM_4. It has been

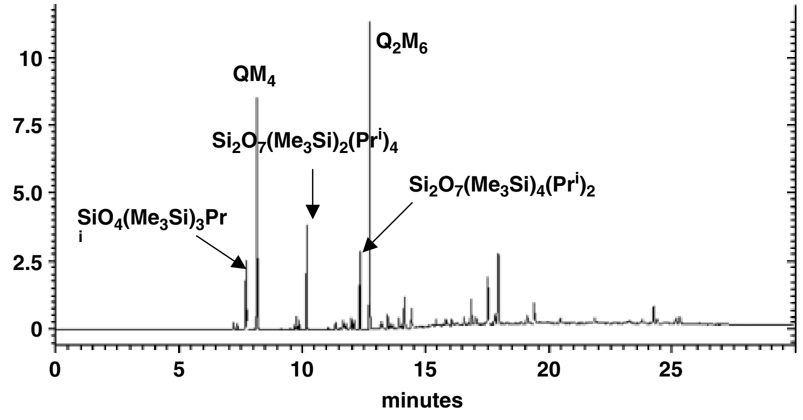

Figure 4. GC of Q_xM_y products (mainly Q_2M_6) obtained from TMS of tailings.

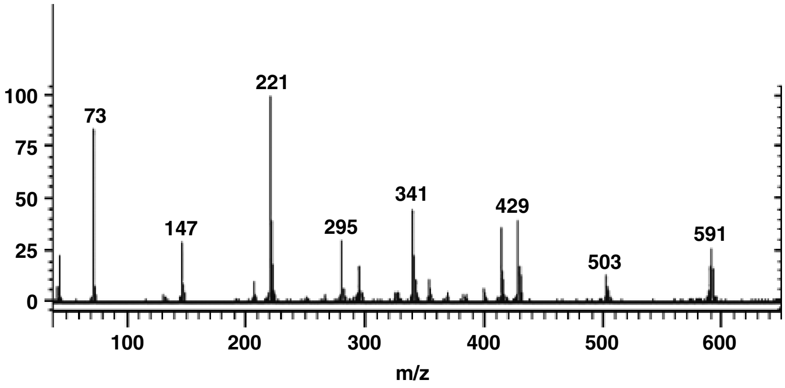

Figure 5. Mass spectra of Q_2M_6 from TMS reaction of tailings.

reported that QM_4 is normally present due to deoligomerization reactions of longer chains.[13] The results are clear evidence for the synthesis of Q_2M_6 from anorthite in the tailings.

Labradorite

Labradorite is an aluminosilicate, also called calcic anorthite, and this phase is present in the sample as the main component. Quartz is the other important phase. The results of the TMS reaction are summarized in Table 1. The best results were obtained after 72 h of reaction. According to GC analysis (Fig. 6), the main product was Q_2M_6 (retention time 13 min). The labradorite sample was used as the silicate most comparable to anorthite with the main purpose of illustrating the reactivity of a similar silicate to the TMS reaction. Labradorite, because of its composition, is less soluble in HCl than anorthite, but soluble enough to be a good candidate for the TMS reaction.

Augite

During the chemical characterization of a silicate ore from an area of (Saltillo State in North Mexico), its solubility in HCl with very low carbonates in composition was observed. The mineralogical analysis revealed it to be a main phase of the mineral augite, with the presence of orthoclase, albite, quartz and iron oxide. Augite is a pyroxene $Ca(Fe,Mg)Si_2O_6$, with very high potential for being tested in the reaction and also for being a good precursor of Q_2M_6 due to the presence of the anion $(Si_2O_6)^{+4}$. Several conditions were tested in order to optimize the reaction. The best conditions were found at 6 h of reaction time at reflux (68 °C), although higher reaction times were used (12 and 24 h) with no appreciable improvement in the reaction yield (about 30%). This is because some of the augite mineral is trapped in the feldspar and quartz matrix. Such information is obtained from optical microscopy. The size reduction from −180 to −325 mesh does not show appreciable increases in yield. It is important to comment that the sample is even reactive at room temperature, but reflux temperature is needed to reduce the reaction time.

Figures 7 and 8 show the GC/MS with an intense signal at 13 min. This is in agreement with the standard obtained from TMS of olivine and the anorthite from tailings. Similar information came from ^{29}Si NMR in solution (signals at 8 and −106 correspond to Q_2M_6 and the signals at 4 and −104 ppm to QM_4). A comparative FTIR spectrum (Fig. 9) is useful to

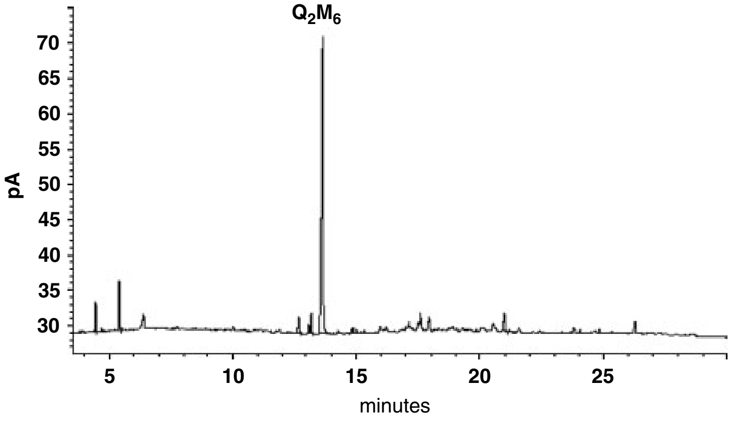

Figure 6. GC of products obtained from TMS of labradorite.

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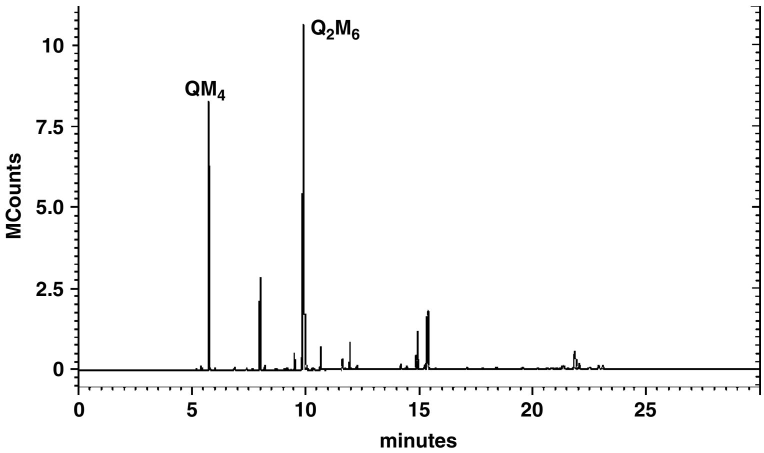

Figure 7. GC of products obtained from TMS of augite.

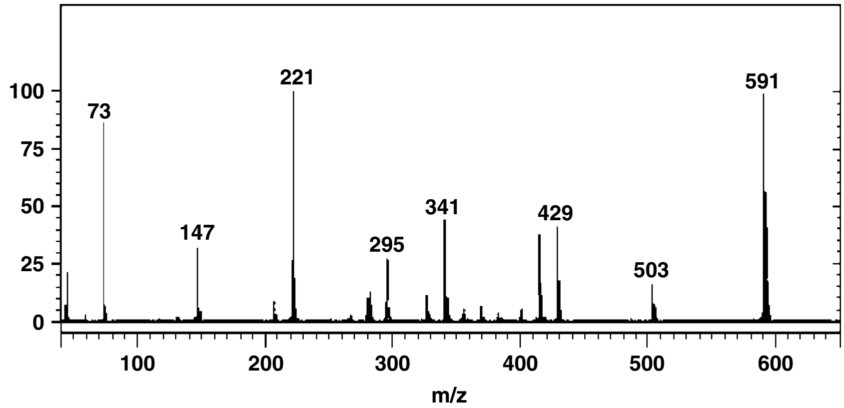

Figure 8. Mass spectra of Q_2M_6 from TMS of augite.

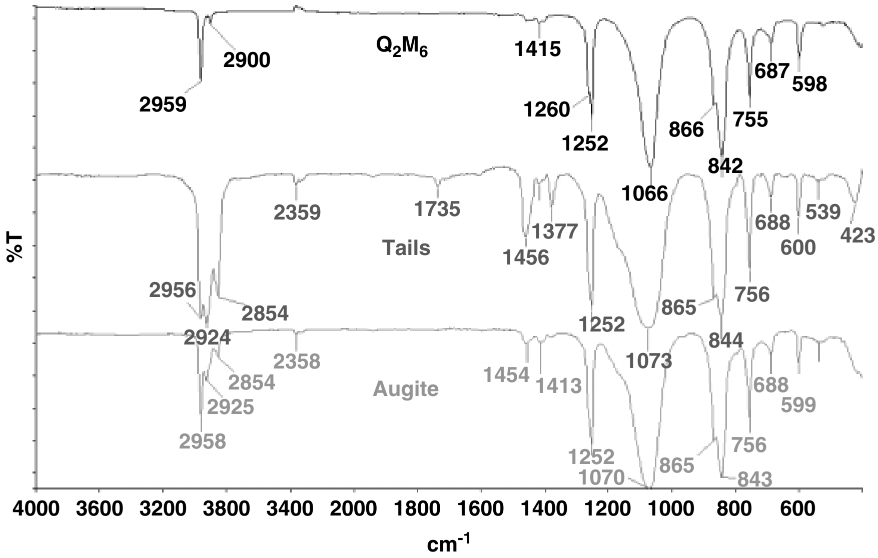

Figure 9. FTIR spectra of Q_2M_6 from TMS of different silicates.

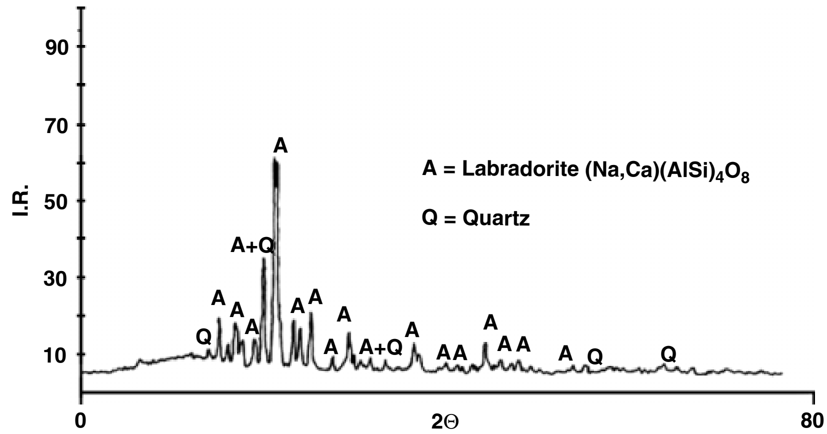

Figure 10. XRD of labradorite before TMS.

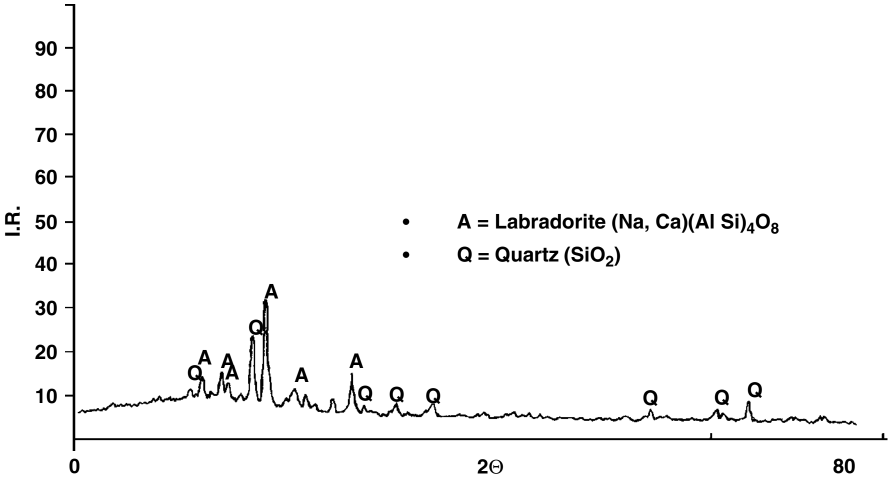

Figure 11. XRD of labradorite after TMS.

illustrate the similarity in products (standard, Q_2M_6 from tailing and from augite). Solution FTIR spectroscopy shows characteristic bands for CH_3 (2959 cm^{-1}), $SiCH_3$ (1252 cm^{-1}), SiOSi (1073 cm^{-1}) and $Si(CH_3)_3$ (756 cm^{-1}).

Studies on the solid residues from the TMS reaction

Because it was considered important to characterize the solid residue from the reaction, each residue was characterized using atomic absorption, XRD, solid-state NMR, thermal analysis and contact angle measurements. A detailed discussion related to each silicate sample is presented.

Labradorite

XRD was used to characterize the material before and after the TMS reaction. For labradorite, Figs 10 and 11 show the diffractograms and it is possible to observe the decay in the signals of the calcic anorthite due to the reactivity of this phase with TMS.

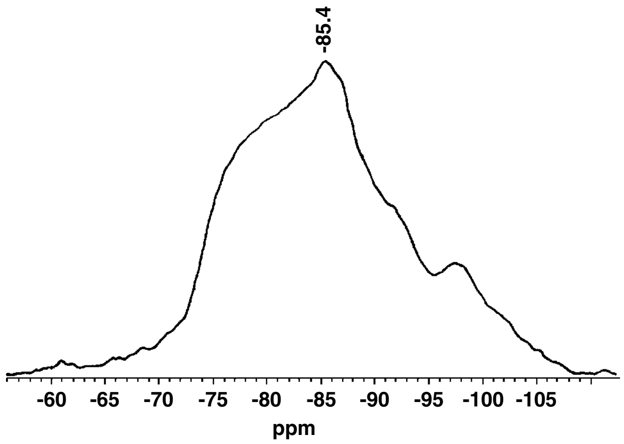

Figure 12. ^{29}Si MAS NMR spectra of labradorite before TMS.

^{29}Si MAS NMR shows some basic modification to the silicon environment as a result of the reaction. Figure 12 illustrates

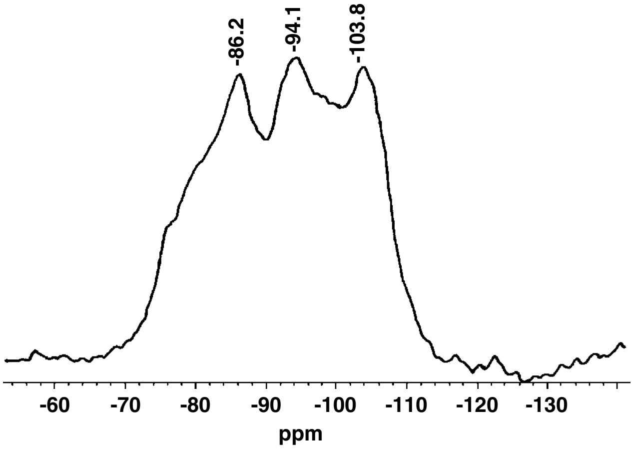

Figure 13. ^{29}Si MAS NMR spectra of labradorite after TMS.

broadened peaks because of the effect of Si, Al disorder. After 72 h of reaction (Fig. 13), the spectra with narrow and resolved peaks indicate a high degree of Si, Al ordering in the feldspar.[12]

Augite

XRD diffraction patterns before and after the TMS reaction (Figs 14 and 15) show, as expected, changes in the augite phase (significant decrease in intensity). The identified phases were the pyroxene-type augite $Ca(Fe,Mg)Si_2O_6$, an orthoclase feldspar ($KAlSi_3O_8$), a feldspar from plagioclases or albite ($NaAlSi_3O_8$), and quartz. Other possible phases in this mineral are gypsum ($CaSO_4 \cdot 2H_2O$) and calcite.

The best way to identify the main phase (augite) is by solid-state MAS NMR ^{29}Si. As can be seen in Fig. 16, there is a signal at −80 ppm in the region typical for inosilicates

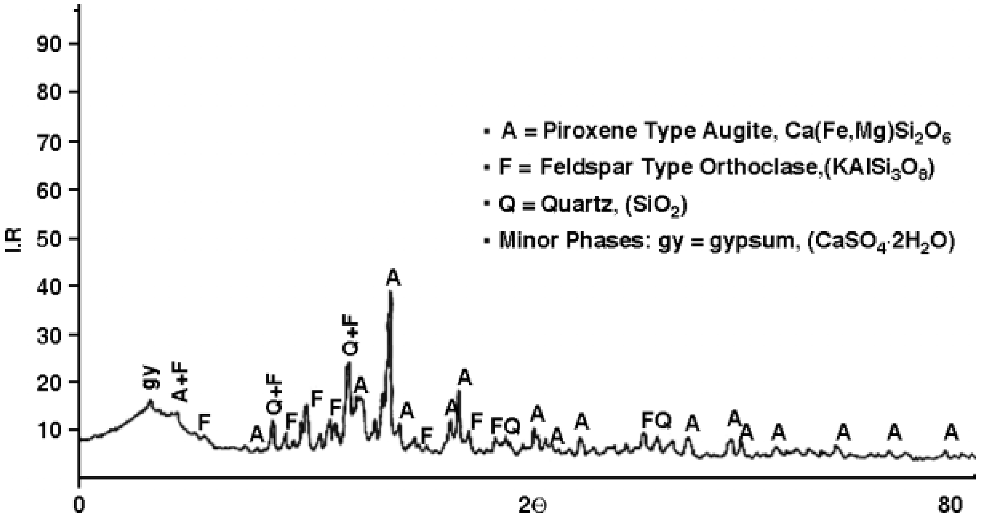

Figure 14. XRD of augite before TMS.

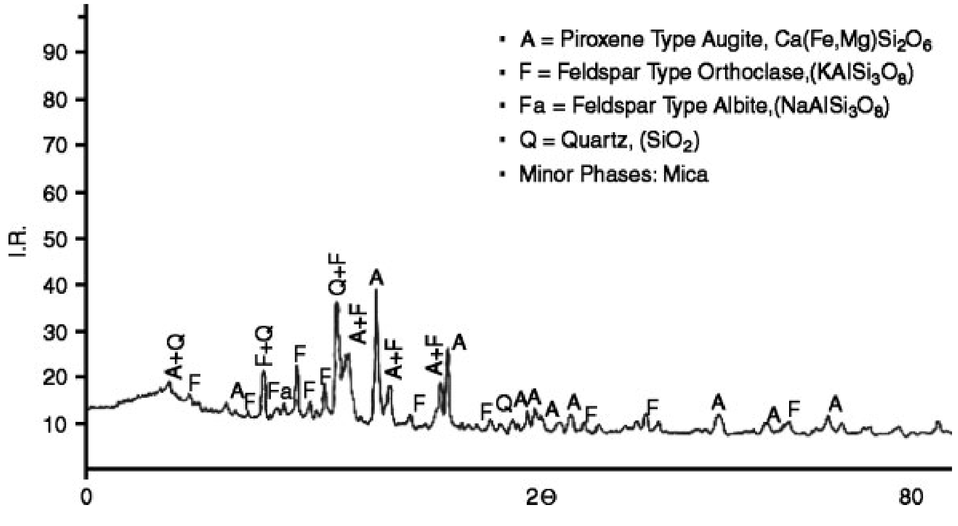

Figure 15. XRD of augite after TMS.

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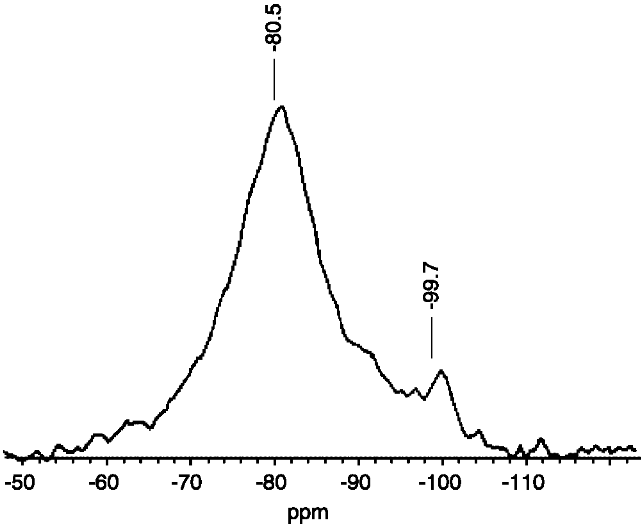

Figure 16. ^{29}Si MAS NMR spectra of augite before TMS.

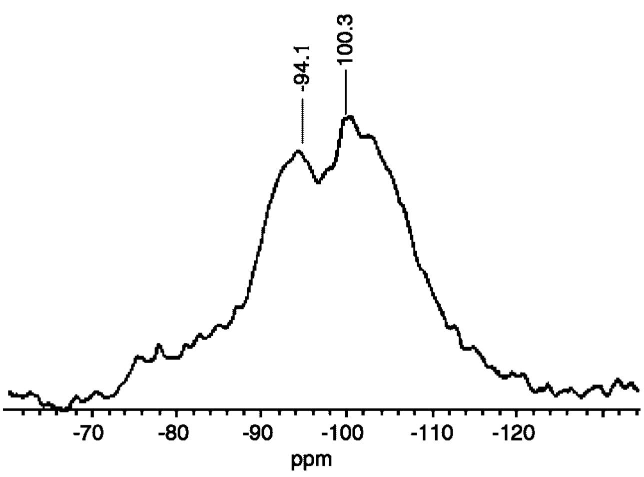

Figure 17. ^{29}Si MAS NMR spectra of augite after TMS.

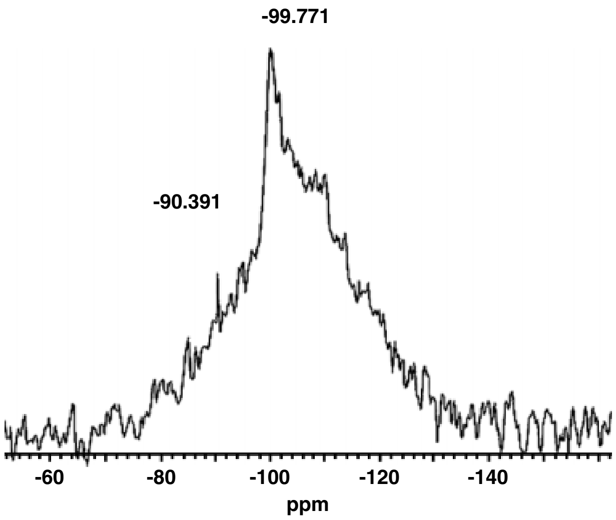

Figure 18. ^{29}Si MAS NMR spectra of tailings before TMS.

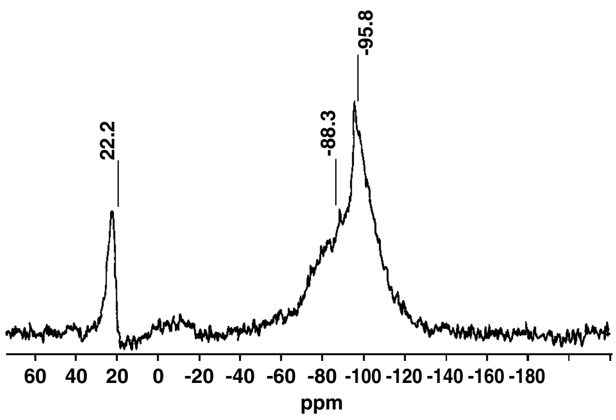

Figure 19. ^{29}Si CPMAS NMR spectra of hydrophobic solid residue from TMS.

(quite similar to Wallostonite[12]) and after the TMS reaction this signal almost disappears (Fig. 17). The signal in the Q^3 region (feldspars) is now more intense.

Tailings

The solid residue was studied previously by XRD and other techniques, and according to these,[9] the main mineralogical phases present are quartz, albite, orthoclase feldspars, calcite, anorthite and chlorite. The solid-state ^{29}Si MAS NMR spectra of tailings before TMS reaction are shown in Fig. 18. Two main signals are observed at −90.3 and −99.7 ppm. After the reaction, a sharp signal at −99.5 ppm is observed. The ^{29}Si MAS NMR spectra suggest that modification occurs as a result of the feldspars' reactivity with TMS (mainly anorthite) to form Q_2M_6.

Hydrophobic character in solid residues

All the solid residues from the TMS reactions have hydrophobic properties due to the incorporation of Me_3SiO- groups at the solid surfaces. The ^{29}Si CPMAS NMR spectra (Fig. 19) give a signal at 18.7 ppm assigned to M groups attached to the surface and the corresponding Q core. The hydrophobic character is lost at 450–500 °C. From DRIFT analysis, it is possible to follow the elimination of bands at CH_3 (2960 cm^{-1}), $SiCH_3$ (1265 cm^{-1}) and $Si(CH_3)_3$ (756 cm^{-1}). More precise information was obtained from DTA analysis (Fig. 20). The mass loss assigned to Me_3SiO- groups occurs from 435 to 600 °C, so the solid is thermally stable to 435 °C. Contact angle measurements were 80° as the average value obtained from the different solid residues after the TMS reactions.

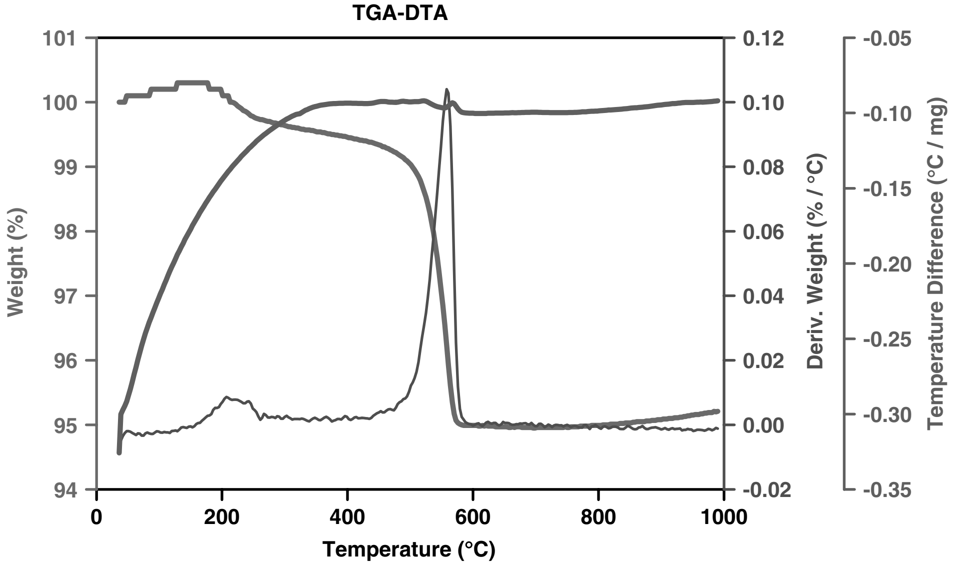

Figure 20. DTA analysis of hydrophobic solid residue from TMS.

CONCLUSIONS

Samples of three complex natural silicates were used as sources of the oligosiloxane Q_2M_6. This oligomer is obtained as the main product in each case. We have demonstrated that in tailings from the flotation process of gold and silver sulfide mineral benefication, the most reactive phase is anorthite, which is present in regular yield and soluble enough in HCl solutions to be a good candidate for the TMS reaction. However the complex mineral, which contains augite as the main phase, is also a natural source useful for this reaction, although it is important to consider the variable ore composition. In the case of labradorite, it was useful to verify the reactivity to TMS reaction of a mineral quite similar to anorthite. We can consider it as a natural standard without the complexity of the complex silicate (tailings) where anorthite is found.

The hydrophobic character of the solid residues after the TMS reaction due to the incorporation of Me_3SiO- groups to the surface is lost around 600 °C. Such solids present thermal stability until 435 °C.

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