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Preparation of silicas combined with optically active organic compounds: optical resolution of metal chelate complexes on the silica composites

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Abstract

A new type of optically active organic–inorganic composite was prepared by a sol–gel method in which tetraethoxysilane is hydrolysed after replacing the ethoxy group with D-lactose or L-tartaric acid, and compared in the optical resolution of tris(2,4-pentanedionato)metal complexes with conventional silica composites (kneading with L-lactose and impregnation with an L-lactose or L-tartaric acid solution) and the organic compounds themselves. The sol–gel composites showed much higher optical resolution abilities than the conventional materials and, in addition the optically active organic compounds could not resolve the racemate under similar conditions. The high resolution ability of the sol–gel composites was deduced to come from the combined effect of silica and a dispersed optically active organic compound.

1. Introduction

Many composites made up of silica and an optically active organic compound have been prepared and used in chromatography [1–13]. However, they are mostly limited to two classes, one in which an optically active organic compound is covalently coupled to a functional group pendant on the silica surface through C–Si bonds such as aminopropyl-derivatized silica [1–8], and another in which the silica surface is coated with an optically active compound such as

cellulose [9–13]. In these composites, silica is used as a support to fix an optically active compound and no further role other than as the support is expected.

We considered that a composite incorporating an optically active compound in the skeleton structure or matrix network of silica should have different properties to a composite bearing an optically active compound on the silica surface, and might show certain synergism of the combined effect of the adsorptive ability of silica and the molecular recognition ability of an optically active compound in separation. Because organic–inorganic composites obtained by combining

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organic and inorganic compounds at a molecular level not only show properties of both the organic and inorganic parts but often also entirely new properties [14].

Here we report the incorporation of optically active organic compounds such as D-lactose, D-glucose, L-tartaric acid and L-malic acid in silica by a sol-gel procedure via the substitution of the ethoxy group of tetraethoxysilane (TEOS) and the optical resolution of tris(2,4-pentanedionato)metal complexes $[M(\text{acac})_3]$ with the optically active organic-silica composites obtained by the sol-gel procedure.

2. Experimental

2.1. Preparation of an optically active organic compound-silica composite

D-Lactose-silica (D-lac-SiO₂)

TEOS (0.6 mol) was mixed with D-lactose (0.04–0.2 mol) in ethanol (100 g) in the presence of acetic acid (0.2 mol) at 80°C for 2 h. To the solution, water (1.2–5.6 mol) was added at the same temperature and allowed to stand for 6–8 h to give a gel. The gel was dried at 80°C under reduced pressure.

D-Glucose-silica (D-glu-SiO₂), D-sorbitol-silica (D-sor-SiO₂) and D-fructose-silica (D-fru-SiO₂)

These three composites were prepared in a manner identical with the above, but in all the preparations the saccharide:TEOS and water:TEOS ratios were only 0.2:0.6 and 2.0:0.6 (mol/mol), respectively.

L-Tartaric acid-silica (L-tart-SiO₂), L-malic acid-silica (L-mal-SiO₂) and L-mandelic acid-silica (L-mand-SiO₂)

These composites were also prepared in a manner identical with the above, but the water:TEOS ratio was always 2 (mol/mol), and the hydroxycarboxylic acid:TEOS ratio was 0.1–0.8:0.6 for L-tart-SiO₂, 0.27:0.40 for L-mal-SiO₂ and 0.40:0.80 for L-mand-SiO₂.

Kneading D-lac-SiO₂, impregnation D-lac-SiO₂ and L-tart-SiO₂

For comparison, D-lac-SiO₂ and L-tart-SiO₂ were also prepared by conventional methods such as kneading and impregnation procedures, in which TEOS was first hydrolysed to silica in the presence of acetic acid, and the silica was kneaded with D-lactose or impregnated with a solution of D-lactose or L-tartaric acid.

In all of the silica composites prepared, the content of the optically active compound was checked by thermogravimetric-differential thermal analysis (TG-DTA) and the optically active compound:SiO₂ ratio was found to be almost equal to the optically active compound:TEOS ratio used in the preparation.

2.2. Packing and elution

All the composites were ground to powders below 200 mesh. Each of the composites was slurried with *n*-hexane and packed in a glass tube (6 mm I.D.). A small amount of a benzene solution of a racemic $M(\text{acac})_3$ was placed on a column and eluted with *n*-hexane-benzene, *n*-hexane-benzene-1,4-dioxane or *n*-hexane-benzene-methyl ethyl ketone (MEK).

2.3. Measurement and characterization

The X-ray diffraction (XRD) patterns were recorded on a MAC Science MXP 18 instrument using Cu K α radiation with a nickel filter. TG-DTA was carried out on a MAC Science TG-DTA 2100 instrument in air. The circular dichroism (CD) spectra were recorded on a JASCO J-600 spectropolarimeter.

3. Results and discussion

3.1. Saccharide-silica composites

Table 1 shows the molecular circular dichroisms ($\Delta\epsilon$) of the first fractions of $\text{Co}(\text{acac})_3$ eluted from the D-lactose monohydrate, kneading, impregnation and sol-gel D-lac-SiO₂ columns with *n*-hexane-benzene-1,4-dioxane

Table 1

Optical resolution of Co(acac)₃ with different D-lac–SiO₂ composites and D-lactose monohydrate

	D-lac:SiO ₂ (mol/mol)	Δε (546 nm) ^a	Column length × I.D. (mm)
D-Lactose monohydrate	∞	0.3 ^b No resolution	900 × 30 380 × 6
Kneading D-lac–SiO ₂	0.1:0.3	No resolution	400 × 6
Impregnation D-lac–SiO ₂	0.1:0.3	No resolution	400 × 6
Sol-gel D-lac–SiO ₂ ^c	0.1:0.3	1.3	370 × 6

Eluent: *n*-hexane–benzene–1,4-dioxane(45:45:10).^a The first fraction eluted.^b Ref. [15].^c H₂O:TEOS = 1.0:0.3.

(45:45:10, v/v/v). Under the conditions used here, kneading and impregnation D-lac–SiO₂ composites and D-lactose monohydrate did not give the optically active complex, and only sol-gel D-lac–SiO₂ partially resolved the racemate into the optically active forms. The resolution ability of sol-gel D-lac–SiO₂ was found to be much higher than those of the others, taking into account the length and diameter of the columns. This suggests that the sol-gel method is suitable for drawing out certain combined effects of organic and inorganic compounds.

The properties of the materials obtained by the sol-gel method are considerably affected by

the preparation conditions, especially the amount of water added in preparing a sol and gel. Table 2 shows the effects of the amounts of water and D-lactose used in preparing D-lac–SiO₂ composite sols and gels on the optical resolution of Co(acac)₃ with the composites, including the effect of the eluents. When the amount of water added increased, the resolution ability of D-lac–SiO₂ initially increased, but decreased again at H₂O:TEOS ratios over 3.33. As for the amount of D-lactose in the composite, the resolution ability increased with increasing D-lactose:TEOS ratio, but it was very difficult to obtain a D-lac–SiO₂ gel at the ratios over 1:3 when the

Table 2

Optical resolution of Co(acac)₃ with sol-gel D-lac–SiO₂ composites

No.	Preparation conditions			Eluent (volume ratio)	Δε (328 nm) ^a	Flow-rate (ml/r)
	Lactose (mol)	TEOS (mol)	H ₂ O (mol)			
1	0.1	0.3	0.6	<i>n</i> -Hexane–benzene–1,4-dioxane (45:45:10)	–1.4	0.008
2	0.1	0.3	1.0	<i>n</i> -Hexane–benzene–1,4-dioxane (45:45:10)	–21.6	0.011
3	0.1	0.3	1.4	<i>n</i> -Hexane–benzene–1,4-dioxane (45:45:10)	–5.8	0.043
4	0.1	0.3	2.2	<i>n</i> -Hexane–benzene–1,4-dioxane (45:45:10)	–6.7	0.025
5	0.1	0.3	2.6	<i>n</i> -Hexane–benzene–1,4-dioxane (45:45:10)	No resolution	0.069
6	0.1	0.3	2.8	<i>n</i> -Hexane–benzene–1,4-dioxane (45:45:10)	No resolution	0.039
7	0.1	0.3	1.0	<i>n</i> -Hexane–benzene–1,4-dioxane (45:45:10)	–21.4	0.009
8	0.08	0.3	1.0	<i>n</i> -Hexane–benzene–1,4-dioxane (45:45:10)	–9.8	0.007
9	0.04	0.3	1.0	<i>n</i> -Hexane–benzene–1,4-dioxane (45:45:10)	–4.3	0.003
10	0.02	0.3	1.0	<i>n</i> -Hexane–benzene–1,4-dioxane (45:45:10)	–3.5	0.001
11	0.1	0.3	1.0	Benzene–1,4-dioxane (90:10)	–9.4	0.005
12	0.1	0.3	1.0	<i>n</i> -Hexane–benzene–acetone (45:45:10)	–21.6	0.004
13	0.1	0.3	1.0	<i>n</i> -Hexane–Benzene–MEK (45:45:10)	–49.2	0.005
14	0.1	0.3	1.0	<i>n</i> -Hexane–benzene– <i>t</i> -BuOH (44:44:12)	–6.2	0.004
15	0.1	0.3	1.0	<i>n</i> -Hexane–benzene–diethyl ether (44:44:12)	No resolution	Fast
16	0.1	0.3	1.0	<i>n</i> -Hexane–benzene (50:50)	No elution	Very slow

^a The first fraction eluted.

H₂O:TEOS ratio was kept 3.33. Hence a D-lactose:TEOS:H₂O ratio of 0.1:0.3:1 in the preparation of the composite seems to be the optimum to obtain a D-lac-SiO₂ with high recognition ability for the chirality of Co(acac)₃. As for the eluent and its flow-rate, mixed solutions such as *n*-hexane–benzene–1,4-dioxane, *n*-hexane–benzene–acetone and *n*-hexane–benzene–methyl ethyl ketone were suitable as the eluents, and at the flow-rates of the eluents listed in Table 2 it was difficult to find some special relationship between the flow-rate and $\Delta\epsilon$ or the optical resolution, because the D-lac:TEOS ratio was variable when $\Delta\epsilon$ increased with an increase in flow-rate.

Table 3 shows the optical resolution of Co(acac)₃ with sol-gel D-glu-SiO₂, D-sor-SiO₂ and D-fru-SiO₂ prepared under conditions similar to the optimum preparation conditions for sol-gel D-lac-SiO₂. These three composites could resolve Co(acac)₃ partially as well as sol-gel D-lac-SiO₂, but only D-fru-SiO₂ shows opposite chirality recognition.

In the above resolution experiments, the performance of the composites in the optical resolution of Co(acac)₃ often varied with the drying conditions before use or the moisture in the composites. This seems to be due to the water in the composites blocking the interaction of Co(acac)₃ with the saccharide and silica parts.

Figs. 1 and 2 show the XRD patterns of silica, D-lac-SiO₂, D-lactose monohydrate, D-fru-SiO₂, D-sor-SiO₂ and D-glu-SiO₂. With sol-gel D-lac-SiO₂ composites, as the amounts of D-lactose (Fig. 1b–d) and water (Fig. 1d–f) used increased, the peaks due to D-lactose crystals

became stronger and sharper. The XRD peaks of kneading and impregnation D-lac-SiO₂ composites are also as sharp as those of D-lactose monohydrate. These results indicate that the dispersibility of D-lactose in the sol-gel composites decreases with increase in the amounts of D-lactose and water used, and then the dispersibility of D-lactose in the kneading and impregnation composites is basically low, because generally crystals show sharper and stronger XRD peaks with increase in size. As D-lactose dissolves easily in water and slightly in ethanol, it seems strange that the dispersibility of D-lactose decreases with increase in the amount of water used. However, it is in fact natural because the C–O–Si bond formed by replacing the ethoxy group of TEOS with D-lactose is easily hydrolysed with a large amount of water and D-lactose tends to come out of the network of silica. This suggests that sol-gel D-lac-SiO₂ composites prepared with a large amount of water would be similar in properties to impregnation D-lac-SiO₂ composites. Actually, the sol-gel D-lac-SiO₂ composites prepared with a large excess of water (e.g., Nos. 5 and 6 in Table 1) were similar in their XRD and TG–DTA patterns and in the resolution behaviour of Co(acac)₃ to the impregnation D-lac-SiO₂ composite, and also to the kneading composite. Impregnation and kneading D-lac-SiO₂ composites showed simple combined patterns of the individual TG–DTA curves of D-lactose and silica, but sol-gel D-lac-SiO₂ composites prepared with H₂O:TEOS ratios below 7 showed different patterns. As for the other saccharide–silica composites, D-glu-SiO₂ composite showed slightly broader XRD peaks, and

Table 3
Optical resolution of Co(acac)₃ with sol-gel saccharide-SiO₂ composites

Composite	Eluent (volume ratio)	$\Delta\epsilon$ (328 nm) ^a
D-lac-SiO ₂	<i>n</i> -Hexane–benzene–MEK (45:45:10)	–49.2
D-glu-SiO ₂	<i>n</i> -Hexane–benzene (50:50)	–21.5
D-sor-SiO ₂	<i>n</i> -Hexane–benzene (50:50)	–19.5
D-fru-SiO ₂	<i>n</i> -Hexane–benzene (50:50)	+61.7

Saccharide:SiO₂ = 1:3; H₂O:TEOS = 1:0.3; column, 400 mm × 6 mm I.D.

^a The first fraction eluted.

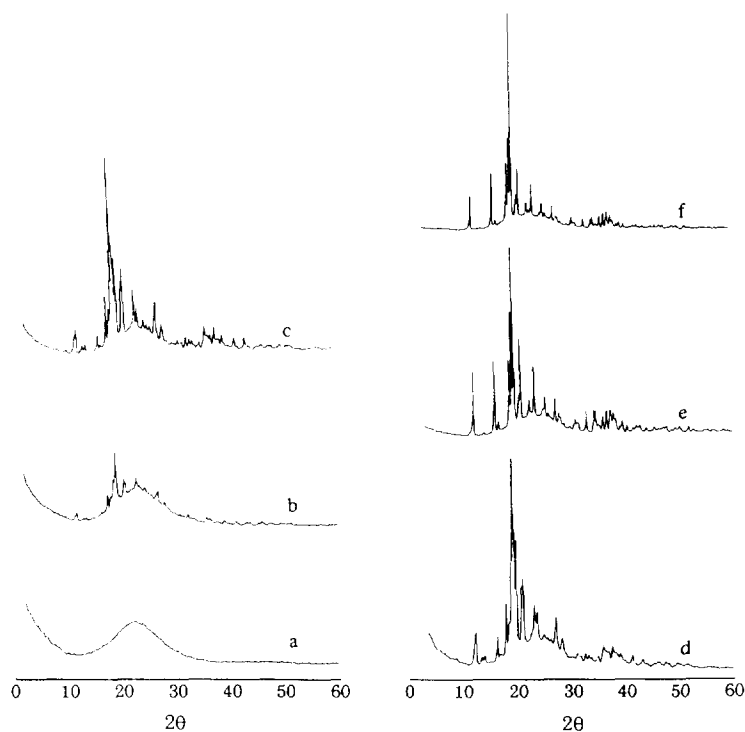


Fig. 1. XRD patterns of sol-gel D-lactose-silica composites and silica. (a) SiO_2 , D-lac-SiO₂; (b) lac:TEOS = 0.02:0.3, H₂O:TEOS = 1:0.3; (c) lac:TEOS = 0.08:0.3, H₂O:TEOS = 1:0.3; (d) lac:TEOS = 0.1:0.3, H₂O:TEOS = 1:0.3; (e) lac:TEOS = 1:0.3, H₂O:TEOS = 2.2:0.3; (f) lac:TEOS = 0.1:0.3, H₂O:TEOS = 2.8:0.3.

both D-sor-SiO₂ and D-fru-SiO₂ composites did not show any clear XRD peaks due to the respective saccharide crystals. However, D-sorbitol, D-fructose and D-glucose could be easily extracted from the respective composites by washing them with water. Hence, it is considered that D-sor-SiO₂ and D-fru-SiO₂ composites do not have D-sorbitol and D-fructose crystals of sufficient size to diffract X-rays, and it is found that D-sorbitol, D-fructose and D-glucose are more dispersed than D-lactose in the composites. The dispersion of saccharides in the composites also seems to be closely related to their solubilities in ethanol, because a large amount of ethanol is produced by the hydrolysis of TEOS or in the sol-gel process, and the solubility of the saccharides used here decreases in the order D-sorbitol \approx D-fructose \gg D-glucose \gg D-lactose [16].

From the above results, it is roughly concluded

that a composite in which a saccharide is highly dispersed shows a higher recognition ability for the chirality of Co(acac)₃ and the high dispersion is the reason why, in the optical resolution of Co(acac)₃, the sol-gel D-lac-SiO₂ composite is superior to the corresponding kneading and impregnation composites.

3.2. Hydroxycarboxylic acid-silica composites

Table 4 shows the results of the optical resolution of Co(acac)₃ with the three sol-gel composites L-tart-SiO₂, L-mal-SiO₂ and L-mand-SiO₂, prepared under similar conditions. Under the conditions used here, L-tart-SiO₂ showed a much higher resolution ability than the other two, as can be seen from the comparison of the $\Delta\epsilon$ values.

Thus, the preparation conditions for L-tart-SiO₂ were investigated in detail. Table 5 gives

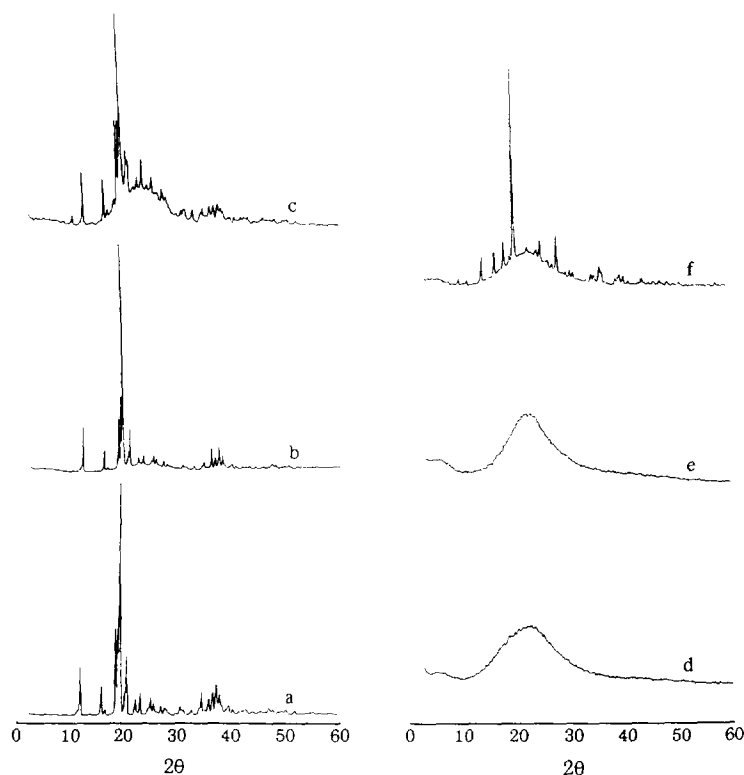


Fig. 2. XRD patterns of saccharide-silica composites and D-lactose monohydrate. (a) D-lactose monohydrate. Saccharide-SiO₂ (saccharide:SiO₂ = 0.1:0.3); (b) kneading D-lac-SiO₂; (c) impregnation D-lac-SiO₂; (d) sol-gel D-fru-SiO₂; (e) sol-gel D-sor-SiO₂; (f) sol-gel D-glu-SiO₂.

the $\Delta\epsilon$ values of the first fractions of Co(acac)₃ eluted from L-tartaric acid and different L-tart-SiO₂ columns with *n*-hexane-benzene (1:1, v/v). With L-tartaric acid itself, the optically active cobalt complex was not obtained. This might be

Table 4
Optical resolution of Co(acac)₃ with sol-gel L-hydroxycarboxylic acid-silica composites

Composite	$\Delta\epsilon^a$	
	328 nm	571 nm
L-tart-SiO ₂	58.5	-4.7
L-mal-SiO ₂	2.8	-0.1
L-mand-SiO ₂	No elution	

L-Hydroxycarboxylic acid:SiO₂ = 0.2:0.6; column, 400 mm × 6 mm I.D.

^a The first fraction eluted.

due to the high flow-rate (0.298 ml/min) of the eluent, but the rate is comparable to that of sol-gel 8 in Table 5. As for impregnation L-tart-SiO₂, only the composites with L-tart:SiO₂ ratios of 0.33–0.67 gave fractions with low optical activity, and it was difficult to resolve the racemate of the cobalt complex using the composites with higher L-tartaric acid contents with L-tart:SiO₂ ≥ 1, even when the flow-rate of the eluent was slow. On the other hand, with sol-gel L-tart-SiO₂, fractions with high optical activity were easily obtained; as the L-tartaric acid content increased, the $\Delta\epsilon$ of the fractions first increased and then decreased after maintaining high values over a wide range of L-tartaric acid content. In addition, even when the tartaric acid content was very high (L-tart:SiO₂ = 1.33) and the flow-rate of eluent was high (0.270 ml/min), the fraction showed weak optical activity. These

Table 5
Effect of the L-tartaric acid content in L-tart–SiO₂ on optical resolution of Co(acac)₃ with L-tart–SiO₂

	L-tart:SiO ₂ (mol/mol)	Flow-rate (ml/min)	$\Delta\epsilon^*$	
			328 nm	571 nm
Sol-gel 1	0.1:0.6	Very slow	No elution	
Sol-gel 2	0.2:0.6	0.005	58.5	–4.7
Sol-gel 3	0.3:0.6	0.021	50.6	–4.8
Sol-gel 4	0.4:0.6	0.022	61.7	–4.9
Sol-gel 5	0.5:0.6	0.044	64.3	–5.3
Sol-gel 6	0.6:0.6	0.042	61.8	–5.0
Sol-gel 7	0.7:0.6	0.098	28.7	–2.3
Sol-gel 8	0.8:0.6	0.270	4.9	–
Impregnation 1	0.1:0.6	Very slow	No elution	
Impregnation 2	0.2:0.6	0.063	13.7	–1.0
Impregnation 4	0.4:0.6	0.076	4.9	–
Impregnation 6	0.6:0.6	0.110	No resolution	
Impregnation 8	0.8:0.6	0.036	No resolution	
L-Tartaric acid	∞	0.298	No resolution	

Column: 6 mm I.D. \times 400 mm; eluent, *n*-hexane/benzene (1:1 v/v).

* The first fraction eluted.

results clearly show that sol-gel L-tart–SiO₂ composites have higher recognition abilities than L-tartaric acid and impregnation L-tart–SiO₂ composites for the chirality of Co(acac)₃, and also a synergistic effect appears in the chirality recognition of L-tart–SiO₂ composites if L-tartaric acid and silica are combined by the sol-gel technique.

Fig. 3 shows CD spectra of the fractions of Co(acac)₃ and Cr(acac)₃ eluted from the columns packed with sol-gel L-tart–SiO₂ (L-tart:SiO₂ = 0.33). Spectra a₁ and b and spectrum a_f show CD patterns characteristic of Δ - and Λ -Co(acac)₃, respectively [17,18], and spectrum c also shows the characteristic pattern of Δ -Cr(acac)₃ [19,20]. Thus, the Λ configuration of the metal chelate complexes is found to interact more strongly than the Δ configuration with L-tart–SiO₂. Although the maximum $\Delta\epsilon$ values of the optically pure Co(acac)₃ are still unknown, the highest $\Delta\epsilon$ value at 571 nm which has been reported so far is around 6. On the other hand, the $\Delta\epsilon$ value at 535 nm of the optically pure Δ -(+)₅₈₉-Cr(acac)₃ is known to be –4.5 [19,20] and this value is consistent with that of the first fraction of Cr(acac)₃ obtained with the 1400 mm

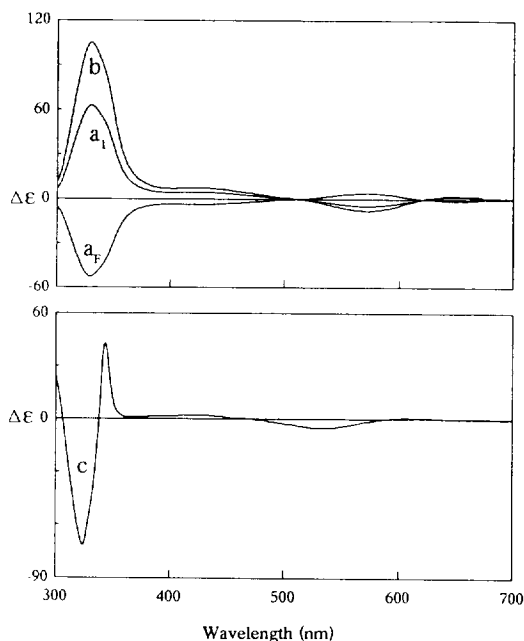


Fig. 3. CD spectra of M(acac)₃ eluted from sol-gel L-tart–SiO₂ with L-tart:SiO₂ = 0.33. Co(acac)₃: (a₁) first fraction by 400-mm column; (a_f) final fraction by 400-mm column; (b) first fraction by 1400-mm column. Cr(acac)₃: (c) first fraction by 1400-mm column.

Table 6
Optical resolution of $M(\text{acac})_3$ with sol-gel L-tart-SiO₂ composite

Co(acac) ₃ Δε ^a		Cr(acac) ₃ Δε ^a		
328 nm	571 nm	323 nm	343 nm	535 nm
103.9	-8.6	-72.2	44.1	-4.4

L-tart:SiO₂ = 0.2:0.6; column 1400 mm × 6 mm I.D.

^aThe first fraction eluted.

column of sol-gel L-tart-SiO₂ (Table 6). Hence it is concluded that the 1400 mm column of sol-gel L-tart-SiO₂ can give the optically pure isomer of Cr(acac)₃, and deduced that the Δε values, -8.6 at 571 nm and 103.9 at 328 nm of the first fraction of Co(acac)₃ obtained by the 1400 mm column, correspond to the Δε values of optically pure Δ-Co(acac)₃.

Fig. 4 shows the XRD patterns of sol-gel L-mand-SiO₂, L-mal-SiO₂ and L-tart-SiO₂ composites. The first shows weak peaks due to L-mandelic acid crystals, but the last two do not show clear XRD peaks. L-Mandelic acid, L-malic acid and L-tartaric acid crystals were easily extracted from the respective composites by washing them with hot ethanol, although their ethylates were also extracted to a small extent. From these facts, it is concluded that L-malic acid and L-tartaric acid are more dispersed than L-mandelic acid in the silicas. This is in harmony with the results in Table 4.

Fig. 5 shows XRD patterns of L-tartaric acid, impregnation L-tart-SiO₂ and sol-gel L-tart-SiO₂ powders. All impregnation L-tart-SiO₂

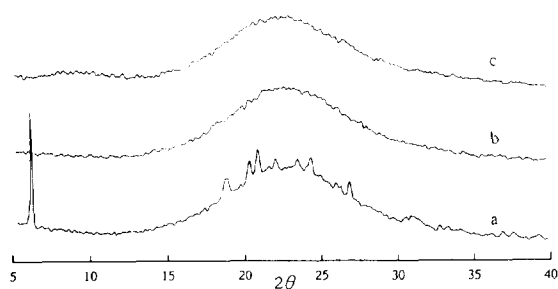


Fig. 4. XRD patterns of sol-gel hydroxycarboxylic acid-silica composites. (a) L-mand-SiO₂; (b) L-mal-SiO₂; (c) L-tart-SiO₂.

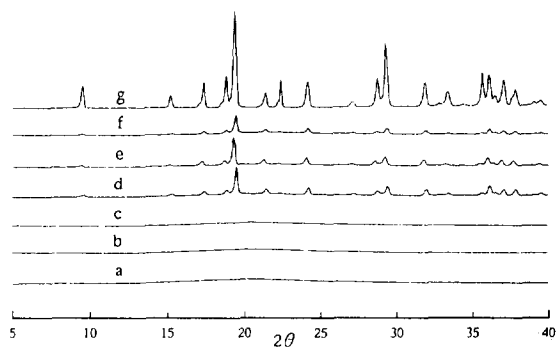


Fig. 5. XRD patterns of L-tartaric acid-silica composites and L-tartaric acid. Sol-gel L-tart-SiO₂: (a) tart:SiO₂ = 0.6:0.6; (b) tart:SiO₂ = 0.4:0.6; (c) tart:SiO₂ = 0.2:0.6. Impregnation L-tart-SiO₂: (d) tart:SiO₂ = 0.6:0.6; (e) tart:SiO₂ = 0.4:0.6; (f) tart:SiO₂ = 0.2:0.6. (g) L-Tartaric acid.

composites listed in Table 5 showed XRD patterns characteristic of L-tartaric acid crystals [21,22] and their TG-DTA curves had an endothermic peak without mass loss around 175°C corresponding to the melting point of L-tartaric acid. In addition, as the L-tartaric acid content in the composites increased, the XRD and TG-DTA peaks became sharper and stronger. These results indicate that, in the impregnation L-tart-SiO₂ composites, L-tartaric acid molecules are easy to aggregate among themselves with order and are dispersed as the crystals. On the other hand, all sol-gel L-tart-SiO₂ composites showed neither clear XRD peaks nor an endothermic peak around 175°C in their TG-DTA curves. This strongly suggests that, in the sol-gel composites, L-tartaric acid is dispersed at the molecular level, because the composites certainly contain a large amount of L-tartaric acid, as stated above. This high dispersion of sol-gel composites would be natural if it is considered that L-tartaric acid bonded to the silicon by substituting the ethoxy group from TEOS is difficult to hydrolyse because L-tartaric acid is a polydentate ligand and can coordinate strongly to a metal ion.

According to the literature [22], L-tartaric acid molecules are held in the crystal structure by a three-dimensional network of O-H-O hydrogen bonds of the usual strength. Thus, when L-tartaric acid is dispersed in the composites as the

crystals, the chance that $\text{Co}(\text{acac})_3$ and $\text{Cr}(\text{acac})_3$ complexes interact with L-tartaric acid is low and the interaction seems to be loose. This is the reason why L-tartaric acid and impregnation L-tart-SiO₂ cannot resolve the metal chelate complexes. In contrast, if L-tartaric acid is dispersed at the molecular level, the metal complexes interact repeatedly and intimately with L-tartaric acid. Alternatively, L-tartaric acid which is dispersed as molecules or clusters in the composites is effective for the chirality recognition or the optical resolution of the metal chelate complexes. As this kind of L-tartaric acid is much more evident in the sol-gel composite than in the corresponding impregnation composite, the former shows a very high performance in the optical resolution of the metal chelate complexes.

4. Conclusions

Saccharides and hydroxycarboxylic acids can substitute alkoxy groups from metal alkoxides. Thus, composites in which optically active organic compounds such as D-fructose, D-sorbitol and L-tartaric acid are highly dispersed in silica can be obtained by a sol-gel procedure using tetraethoxysilane as the silica source.

The composites obtained by the sol-gel procedure show much higher abilities in the optical resolution of tris(2,4-pentanedionato)metal complexes than those obtained by kneading and impregnation procedures, and optically active saccharides and hydroxycarboxylic acids themselves.

The sol-gel procedure is suitable for drawing out the potential of optically active organic compounds for chirality recognition or certain

combined effects of organic and inorganic compounds.

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