

Physical behaviour and composition of low- and high-melting fractions of sediment in canola oil

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The effects of haze composition variation on sedimentation phenomenon in canola oil were studied. Comparisons were made of the physical properties between an oil sediment (S5) and the high- and low-melting fractions obtained by step crystallisation at 32 and 5°C (F32 and F5). The phase transition behaviour and crystalline characteristics of the sediments were examined by differential scanning calorimetry (DSC), hot-stage microscopy, spectrophotometry, and X-ray diffraction (XRD). The compositions of the sediments were determined by GC-MS. The melting behaviour of S5 resembled that of F5, whereas S5 behaved more like F32 in crystallisation. Oils containing F32 clouded much faster than oils containing F5. The interfacial surface free energies and the XRD patterns were similar for the two fractions, suggesting identical crystalline structures. The molecular origin of the varied phase transition behaviour of the sediments was attributed to the composition variation in waxes, particularly the acid component in the materials.

INTRODUCTION

Vegetable oils should be clear and free from sediment formation during storage. Some oils, such as sunflower and cottonseed, contain minor amounts of high-melting substances, which make them prone to sedimentation upon storage if they are not processed properly prior to bottling (Weiss, 1966; Rivarola *et al.*, 1985; Turkulov *et al.*, 1986; Chulu *et al.*, 1989). The nature of sediment in these oils has been determined; the major clouding substances in sunflower seed oil being wax esters of long-chain saturated fatty acids from C20:0 to C28:0 and fatty alcohols from C22:0 to C30:0 (Rivarola *et al.*, 1985). A process, referred to as winterisation, is normally used to remove these substances (Leibovitz & Ruckenstein, 1984).

Canola oil, a genetically modified rapeseed oil, has recently become important in the world oil market due to its nutritional quality (McDonald, 1987). As with other vegetable oils, canola oil must also meet the quality requirement of clarity. In this respect, canola oil does not normally require winterisation, but in recent years, a sedimentation problem has been reported occasionally (Daun & Jeffery, 1991; Hu *et al.*, 1993; Liu *et al.*, 1993). The seriousness of this quality defect varies

from year to year and until now it has not been possible to predict its occurrence. Winterisation, on the other hand, is an expensive process and cannot be used indiscriminately in canola oil if a competitive edge over other oils is to be maintained.

The sedimentation problem in canola oil has recently been studied by several research groups (Daun & Jeffery, 1991; Hu *et al.*, 1993; Liu *et al.*, 1993). Both chemical composition and phase transition behaviour of the sediment were investigated. These workers found the major clouding substances in canola oil were wax esters of fatty acids from C16:0 to C32:0, and fatty alcohols from C16:0 to C32:0, comparable to sunflower seed waxes (Liu *et al.*, 1993). Other substances such as saturated triglycerides, hydrocarbons, free fatty acids, and free fatty alcohols were also reported as minor components (Hu *et al.*, 1993; Liu *et al.*, 1993). In these studies it was reported that sedimentation in oil could not be completely eliminated by removing the sediment as subsequent storage of the oil still resulted in sediment formation (Daun & Jeffery, 1991; Hu *et al.*, 1993; Przybylski *et al.*, 1993). Preliminary experiments indicated that samples of canola oil exhibiting sedimentation may not necessarily contain high amounts of waxes, suggesting other contributing factors.

As with all natural fats, canola sediment is not a homogeneous material, but rather a mixture of various compounds. The physical properties and phase transition

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behaviour of the sediment are therefore dependent on the actual composition, which may vary with variety and environmental conditions during seed development. The relationship between sediment composition and physical behaviour has not been explored. In cocoa butter, which is a mixture of different triglycerides, the physical behaviour is dictated by a singular proportion of the components, resulting in unique melting and crystallisation properties (Schlichter-Aronhime & Garti, 1988).

In our previous study (Liu *et al.*, 1993) on canola oil sediment, the occurrence and physical properties of the sediments crystallised at 32 and 5°C were partly examined. In the present report, canola oil sediment fractions were obtained and their contributions to the overall characteristics of phase transformation of sediment were studied. The phase transition behaviour of the sediment and their low and high melting fractions were further investigated in terms of composition differences between the sediments.

MATERIALS AND METHODS

Preparation of canola oil, sediment and sediment/oil solutions

Refined, bleached and deodorised canola oil and industrial filter cakes were obtained from Western Canadian processors. The filter cakes were collected after filtration of the winterised oil. Canola oil was kept at 0°C for 1 week. The oil was thereafter filtered through filter paper (No. 541, Maidstone, Kent, UK) under vacuum with diatomaceous earth as a filtering aid to remove any solids formed during the cold storage. The filtered oil was used throughout the studies.

Canola oil sediment was extracted with boiling chloroform from the industrial filter cakes. The extract was concentrated with a rotary evaporator to about 30% of the starting volume and stored at 2°C until the sediment settled from the solution. The precipitate was separated from the solution by centrifugation ($16300 \times g$) at 2°C. The isolated material was washed twice with cold petroleum ether (2°C) to remove the residual oil (Przybylski *et al.*, 1993).

Sediment/oil solutions were prepared by dissolving a required amount of sediment in oil on a weight to weight basis or by diluting a standard 2000 ppm sediment/oil solution to the desired concentration.

Fractionation of canola sediment

Canola sediment was fractionated by a step crystallisation technique at two temperatures. An oil solution (0.1%) was heated to 70°C to melt all the crystals and then stored at 32°C until a sediment appeared. The solution was held for further 1 week to allow completion of crystallisation. The sediment was separated from the oil by filtration. The filtrate was then stored at 5°C and the subsequently formed sediment isolated by

centrifugation ($16300 \times g$) and filtration. The yield ratio of the two fractions obtained was about 1:1 by weight. The sediment fractions crystallised at 32 and 5°C, as described above, are referred to as F32 and F5 sediment, respectively.

A portion of the 32°C filtrate was stored at 32°C for an additional period of time. No further sediment was formed in the filtrate, implying a complete removal of all crystallisable components at this temperature.

A sediment was also obtained by storing a sample of the oil solution at 5°C directly. The sediment obtained (referred to as S5) was in effect a mixture of the F32 and F5 fractions.

Composition analysis of sediments

The composition of canola sediments was determined by a TLC-FID (IATROSCAN) procedure (Przybylski & Eskin, 1991). Sediment solutions in benzene at concentration of 5% (w/w) were applied on chromarods III, 0.5 μ l for each rod. The samples were developed in hexane:benzene:acetone:acetic acid (44:30:0.8:0.3, v/v). Contribution of each component was calculated from individual calibration. The fatty acids and alcohols were analysed by GC-MS (Przybylski *et al.*, 1993). The GC instrument used was a Perkin-Elmer gas chromatograph (model 8500 Norwalk, CT, USA) with a capillary column (0.25 mm i.d. \times 50 m) coated with BD-5 (J & W, Folsom, CA, USA). The column temperature was programmed from 175 to 285°C at a rate of 3°C/min. Identification of the compounds was performed using a Finnigan Ion Trap Detector (San Jose, CA, USA) mass spectrometer with the column and conditions as described above for GC. All chemical reagents used were of analytical grade.

Differential scanning calorimetry (DSC), hot-stage microscopy, spectrophotometry, X-ray diffraction, and clouding time measurements

The phase transition behaviour of canola oil sediments was studied by various experimental methods. The melting and crystallisation characteristics of sediments were examined by a thermal analyser (Dupont 9900, Wilmington, DE, USA) equipped with a Dupont 910 DSC cell. Sediments (~3 mg) sealed in DSC pans were heated over the temperature range of 5–100°C at 10°C/min for melting, and cooled at 1°C/min for crystallisation. DSC experiments were also conducted on samples containing various amounts of oil to probe the melting behaviour of sediment in a liquid oil phase. Samples containing various weight fractions of oil were prepared by adding the desired amount of oil into the pans by a microsyringe. An empty pan was used as an inert reference and the DSC instrument was calibrated with indium (Liu *et al.*, 1993). The melting temperature of sediment was taken as the temperature of the DSC peaks. All DSC thermograms reported were normalised on a constant weight basis of sediment solids.

A polarised light microscope (Zeiss, IIRS, Oberkochen, Germany) equipped with a hot stage (Physitemp, Clifton, NJ, USA) was also employed to study the melting and crystallisation behaviour of sediment/oil solutions. Oil samples were enclosed in glass slips and heated or cooled with the hot plate in the temperature range of 5–70°C at a constant rate of 1°C/min. The crystallisation temperature was recorded when the first crystals were observed, whereas the melting temperature was obtained when all the crystals disappeared.

A spectrophotometer (8451a, Hewlett-Packard, Palo Alto, CA, USA) was used to follow the change of intensity of transmitted light through the oil samples during the course of clouding. Oil solutions were placed in a glass cuvette and the samples maintained at a constant temperature of 26°C, by circulating water in the jacket of the cuvette holder. The experimental temperature was so chosen that the oil solutions studied would not cloud instantly but after a period of time. The time for the turbidity development is referred to as induction time (τ) and was estimated from the transmittance/time curves. The interfacial surface free energy between oil and the sediment crystal was derived from the induction times according to the procedure described elsewhere (Liu *et al.*, 1993).

X-ray powder diffraction patterns of sediments were obtained using an X-ray diffractometer (Philips, PW1710, Cincinnati, OH, USA) with $\text{CuK}\alpha$ radiation. The equipment was operated at 40 kV and 40 mA at room temperature.

The time for turbidity appearance (clouding time) was measured isothermally on oils containing various amounts of sediment (25–300 ppm). Oil samples were stored at 22°C, and the time required for the appearance of turbidity, as determined by visual inspection of the oil specimens, was recorded (Liu *et al.*, 1993).

RESULTS AND DISCUSSION

Characteristics of melting and melt crystallisation of sediments

The DSC thermal curves for melting of the canola sediments are shown in Fig. 1. The melting endotherm of the 5°C fraction (F5) was located in a lower temperature range with a peak temperature of $75.2 \pm 0.2^\circ\text{C}$ (Fig. 1a). The 32°C fraction (F32) exhibited a melting peak situated at a higher temperature with a peak at $78.7 \pm 0.2^\circ\text{C}$ (Fig. 1b). The melting enthalpies for these fractions, as determined from the DSC peak areas, were $172 \pm 3 \text{ J/g}$ and $148 \pm 4 \text{ J/g}$ for the F32 and F5, respectively. This can be explained by segregation of components with higher melting enthalpies in the high temperature fraction (Liu *et al.*, 1993).

Figure 1c shows the DSC melting curve for the S5 sediment. Being a mixture of F32 and F5 sediments, this sample exhibited an intermediate melting behaviour between the two fractions. The melting enthalpy was $160 \pm 6 \text{ J/g}$, as would be expected from

the 1:1 yield ratio of the two fractions. However, the peak temperature ($76.3 \pm 0.1^\circ\text{C}$), the conclusion temperature, and the endotherm band for S5 resembled those for F5, suggesting that the low-melting components in S5 sediment played a dominant role in dictating the melting characteristics of the mixture. In a previous study (Liu *et al.*, 1993), we also compared the melting properties of the sediments crystallised at 32 and 5°C. Those materials were relevant to F32 and S5 sediments in the present study but had higher melting enthalpies and narrower DSC peaks. This may reflect the compositional variation of the raw materials. The relevant F5 fraction, however, was not examined in the previous investigation. The finding in the present study further demonstrated the relative contributions of low- and high-melting fractions of the sediment in the melting transition.

Figure 2 shows the corresponding DSC thermograms of melt crystallisation of the sediments. In contrast to the melting curves, the DSC melt crystallisation curves exhibited biphasic transitions. However, the enthalpy of crystallisation was closely matched with that of melting for each sediment. The crystallisation behaviour of F32 and F5 sediments was different. A sharp peak at $75.0 \pm 0.1^\circ\text{C}$ followed by a shoulder was observed for F5 (Fig. 2a), whereas two distinguished peaks at 79.7 ± 0.2 and $76.7 \pm 1^\circ\text{C}$, respectively, were seen for F32 (Fig. 2b). This might suggest a more homogeneous sample in the case of F5 compared with F32. The crystallisation thermal curve of S5 also exhibited two peaks at $78.2 \pm 0.3^\circ\text{C}$ and $74.8 \pm 0.2^\circ\text{C}$, respectively (Fig. 2c), similar to the behaviour of F32.

In the presence of liquid oil medium, the melting characteristics of canola sediments were modified. As the oil fraction increased, the DSC peaks broadened and the melting enthalpies decreased in a similar manner for both fractions. Figure 3 shows the DSC melting peak temperatures as a function of the molar fraction of sediment according to the solubility law; in this case,

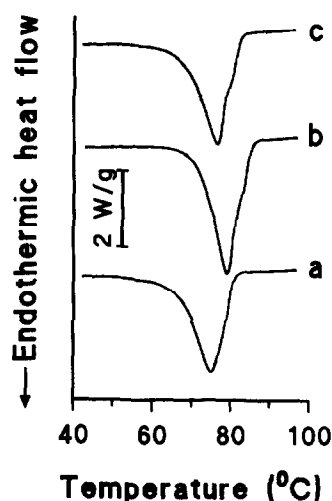


Fig. 1. DSC melting thermograms for canola oil sediments. (a) 5°C fraction (F5); (b) 32°C fraction (F32); and (c) 5°C sediment (S5). DSC heating rate was 10°C/min.

average values for molecular weights of sediment (700) and oil (880) were assumed (Liu *et al.*, 1993). The melting temperature of sediment decreased as the amount of oil in the sample increased. Using the solubility law, enthalpies for the pure sediments were estimated from the slopes of the lines. The derived values were twice the experimental values (297 J/g versus 148 J/g for F5 and 343 J/g versus 172 J/g for F32). However, the ratio of the two enthalpies was similar to that obtained from the ΔH values determined by direct DSC. The discrepancy between the derived and experimental values may be due to the heterogenous nature of sediment. Sediment components that melt at lower than the peak temperature may have considerable lower enthalpies than the average resulting in a lower overall melting enthalpy.

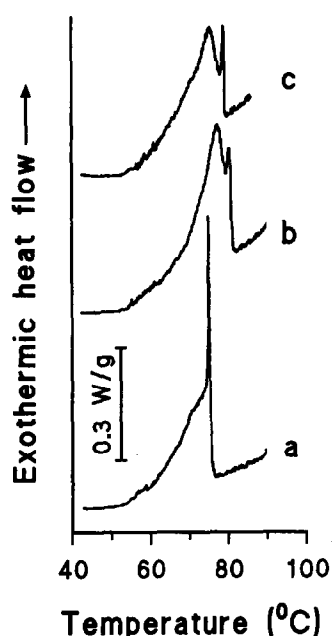


Fig. 2. DSC thermograms of melt crystallisation for canola oil sediments. (a) 5°C fraction (F5); (b) 32°C fraction (F32); and (c) 5°C sediment (S5). DSC cooling rate was 1°C/min.

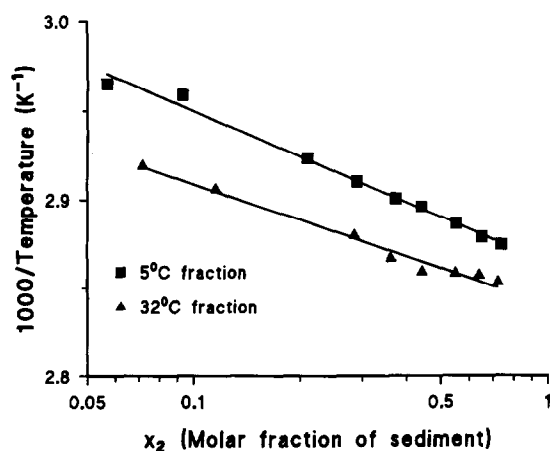


Fig. 3. DSC melting peak temperature as a function of concentration (molar fraction) of sediment in canola oil.

Melting and crystallisation temperatures of sediments in oil

The melting and crystallisation temperatures of sediment in oils that contain very small amounts of sediment (less than 1%) could be readily measured by hot-stage microscopy under polarised light. Knowledge of the relationships between these temperatures and sediment content is important for winterisation where oil is cooled continuously to a final crystallisation temperature and is thereafter brought to a higher temperature for easy filtration. Figure 4 shows these temperatures as a function of sediment content in oil. Both fractions exhibited a very large degree of supercooling of approximately 22°C. The melting and crystallisation temperatures for F32 were about 6°C higher than those for F5.

The phase transition temperatures for S5 sediment are also shown in Fig. 4; the actual data points were not plotted for clarity of presentation. Figure 4 shows the crystallisation temperature for S5 was similar to that for F32, suggesting the high-melting fraction of sediment nucleated in oil first and probably initiated crystallisation. On the other hand, the melting curve for S5 was close to that for F5, indicating the low-melting component was a determining factor in melting. These findings are consistent with the DSC results as shown above where the DSC melt crystallisation of S5 resembled that of F32, while the DSC melting of S5 was comparable to that of F5.

Although the dependence of the melting temperature on sediment concentration can be described by a solubility-law type equation (Eq. 1, Liu *et al.*, 1993), the usefulness of such an equation is limited in practice because it relates the reciprocal of the temperature with the molar fraction of the solute, where the knowledge of an average molecular weight for the solute has to be assumed. A simpler equation is desirable. On the other hand, a quantitative relationship between concentration and crystallisation temperature is needed in many occasions where controlled crystallisation is required (Turkulov *et al.*, 1986; Liu *et al.*, 1993). Using the data

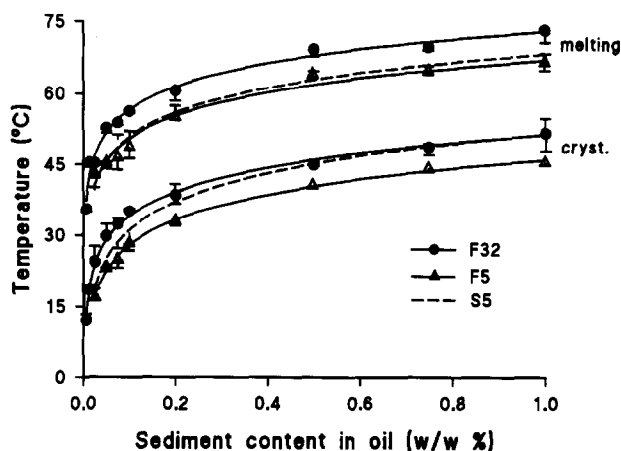


Fig. 4. Microscopic measurements of melting and crystallisation temperatures versus sediment content in canola oil.

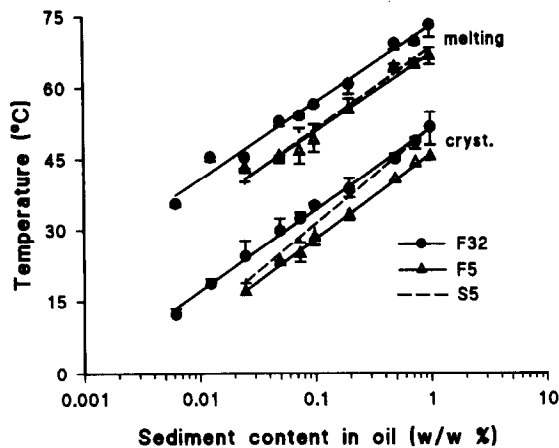


Fig. 5. Relationship between phase transition temperatures and sediment content in canola oil.

in Fig. 4, a general semi-log equation was found to describe adequately the dependence of both melting and crystallisation temperatures on sediment content. This is more clearly shown by Fig. 5, which demonstrated a linear relationship between the transition temperatures and logarithm of sediment content in oil with a coefficient of determination higher than 0.98 for all the regression lines. Such a relation between concentration and melting temperature implies that the solubility of sediment changes with temperature by a constant factor, independent of the absolute concentration of sediment. For example, when temperature increases by 5°C, the solubility of the S5 sediment in oil will always increase by about two fold. A semi-log relation for crystallisation means that decreasing sediment content by a factor decreases the crystallisation temperature by the same degrees.

Interfacial surface free energies of sediment/oil

Figure 6 plots the induction time (τ) of crystallisation as a function of supersaturation (S). Supersaturation is defined as the ratio of sample concentration to the concentration at saturation, which was extrapolated from the melting curves shown in Fig. 5. Figure 6 shows that the induction times fall generally on the same line for both F32 and F5 sediments, suggesting similar solid-liquid surface free energies according to the classical theory of crystallisation. Using the equation derived previously (Liu *et al.*, 1993), the surface free energy between the sediment crystal and oil was estimated to be $10.5 \pm 0.5 \text{ erg/cm}^2$ from the slope of the line. A surface free energy of about half this value was found in an earlier study for sediment isolated from a different batch of filter cakes. A higher surface free energy for the sediment of the present work was indeed expected as the material exhibited a higher degree of supercooling, 22°C compared with 18°C of the previous studies (Liu *et al.*, 1993). However, it is recognised that composition variation of sediment may also contribute to the differences in surface free energy. Any foreign solids present in sediment could reduce the activation

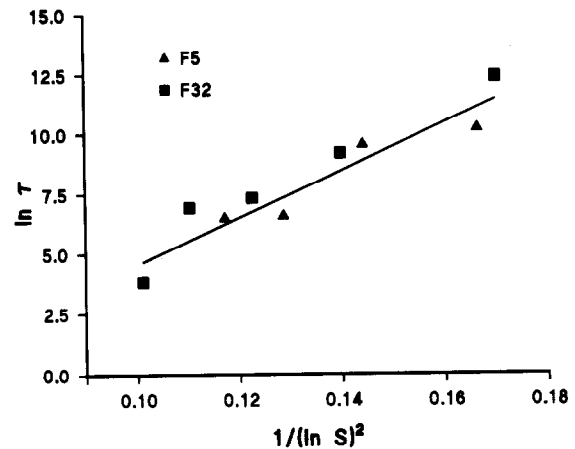


Fig. 6. Induction time (τ) of crystallisation at 26°C as a function of supersaturation (S) of canola oil solutions with various amounts of F5 or F32 sediment.

energy for nucleation, resulting in the differences of calculated value of this physical property (Boistelle, 1988).

Crystalline organisation of sediments

The X-ray diffraction (XRD) patterns of F5 and F32 sediment crystals were identical (the XRD patterns are not shown here). The strongest peaks in the XRD patterns were found at d -spacings of 4.14 and 3.72 Å, respectively. Peaks were also observed at d -spacings of 2.98 and 2.48 Å. These findings are consistent with earlier studies (Liu *et al.*, 1993) which showed that sediments crystallised at different temperatures (materials relevant to F32 and S5 sediments in the present study) had comparable XRD patterns and exhibited similar structural features to crystalline waxes of different origins. The similar XRD patterns obtained for the two fractions also suggested that the observed differences in physical behaviour of the two sediments were not due to different crystal packings.

Concentration dependence of sedimentation in oil

The clouding time in oils that contain various amounts of sediment are shown in Fig. 7. At the same sediment concentration, the clouding time for oils containing F32 was shortest, followed by S5, and oils containing F5 required a longer time to develop turbidity. At 100 ppm sediment level, for instance, the time needed for turbidity appearance was about 1.5 h for the F32 oil solution, whereas 48 h was required for F5 oil.

Figure 7 shows that the dependence of clouding time on sediment concentration followed a log-log relationship. As sediment concentration decreased, the clouding time increased. The increase in clouding time with decreasing sediment concentration for oils containing F5 is more pronounced than for oils containing F32, as indicated by a steeper concentration-time curve for F5 (the slope of the curve for F5 is -3.461 ± 0.291 compared to -2.651 ± 0.156 for F32). Oils containing S5 had a slope value between F5 and F32 (-3.176 ± 0.288).

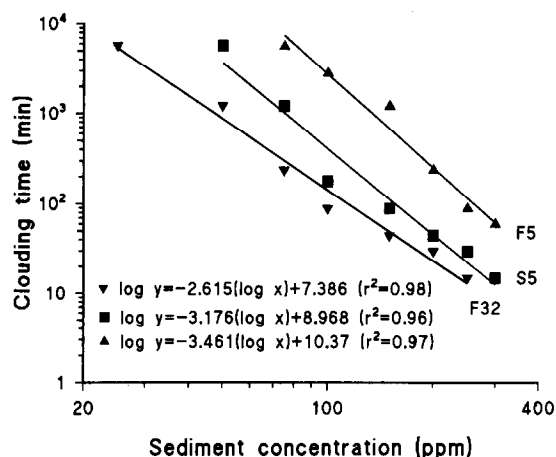


Fig. 7. Relationship between clouding time and sediment concentration in canola oil at 22°C.

Molecular origin of phase transitions of sediment

Table 1 shows that the major components of the fractions (F5, F32) and sediment (S5) were wax esters. Both fractions contained similar amounts of waxes, alcohols and monoglycerides. The content of 1,3-diglycerides in the F5 fraction and S5 sediment were about four times that in the F32 fraction. S5 differed from the two fractions by having slightly lower contents in both wax esters and alcohols.

Polar compounds were found to be present in the sediments. The amount of the polar compounds was higher in F32 than in F5 and S5. The nature of the polar component in the sediments, however, remained unclear. These materials could not be separated or moved by solvents normally used for separating phospholipids (Przybylski & Eskin, 1991). Attempts to separate these components using a very polar solvent mixture such as chloroform:benzene:methanol:water (20:20:50:4, v/v) proved unsuccessful suggesting it was neither glycolipids nor phospholipids. Application of specific phospholipid spray, a modified molybdenum reagent of Zinzadze, also failed to detect any phospholipids or phosphorus-containing compounds (Dittmer & Lester, 1964). Transesterification of the isolated polar materials with methanolic-HCl did not release any fatty acids further suggesting that it was not a lipid component. Similar compounds have been previously reported

Table 1. Composition of high-melting (32°C) and low-melting (5°C) fractions of sediment and the corresponding sediment formed at 5°C (S5) in canola oil (% ± SD)

Component	5°C fraction	32°C fraction	5°C sediment
Wax esters	80.0 ± 0.9	79.5 ± 1.1	77.5 ± 1.1
Alcohols	4.4 ± 0.3	4.6 ± 0.3	3.3 ± 0.3
Monoglycerides	4.1 ± 0.3	4.5 ± 0.3	4.1 ± 0.4
1,3-Diglycerides	5.6 ± 0.5	1.3 ± 0.3	6.3 ± 0.5
Triglycerides	Trace	ND ^a	Trace
Polar compounds	5.9 ± 0.5	10.2 ± 0.6	8.8 ± 0.6

^aND, Not detected.

in canola oil gums (Ratnayake *et al.*, 1985) as possible breakdown or polymeric products from phospholipids. Preliminary elementary analysis showed that these materials were composed of carbon, oxygen and hydrogen, similar to complex carbohydrates.

Fatty acids and alcohols present in the sediment and fractions are shown in Table 2. Significant differences were observed ($P < 0.05$) between the two fractions. The content of very long chain fatty acids, with 24 or more carbon atoms in the chain, was 12% higher in F32 than in F5 (70.4 versus 82.6%). The most abundant fatty acid in F32 fraction was C26:0 compared with C20:0 in F5. The relative proportions of various fatty alcohols in the sediment and fractions were similar with slightly higher contribution of very long chain alcohols in the F32 fraction only. Over 90% of the fatty alcohols were composed of 20 or more carbon atoms in the chain. The presence of longer chain fatty acids and alcohols can have a direct impact on the physical properties of sediments derived from them. An immediate effect will be an increased melting temperature and a lower saturation concentration resulting in a higher sedimentation temperature.

In a heterogeneous system containing components of different melting and crystallisation characteristics, the crystallisation temperature may be expected to influence the composition of the crystals obtained. However, studies of the effect of crystallisation temperature on milkfat fractions by a number investigators have shown very small differences in the composition of the fractions (Grall & Hartel, 1992). Nevertheless, the physical properties of these materials, including thermal properties (melting and crystallisation), were significantly different. Grall and Hartel (1992) suggested that this was

Table 2. Comparison of major fatty acids and alcohols in F5 and F32 sediment fractions and S5 sediment (% w/w) in canola oil

Compound	5°C fraction		32° fraction		5°C sediment	
	Acid	Alcohol	Acid	Alcohol	Acid	Alcohol
12:0	0.05	0.04	0.03	0.02	0.06	0.03
14:0	0.07	0.02	0.03	0.01	0.07	0.02
15:0	0.04	0.05	0.01	0.01	0.05	0.02
16:0	3.03	0.03	1.00	0.01	3.33	0.03
16:1	0.06	—	0.03	—	0.13	—
17:0	0.16	0.23	0.06	0.09	0.13	0.19
18:0	6.65	0.21	2.97	0.09	5.79	0.16
19:0	0.45	0.04	0.28	0.02	0.38	0.04
20:0	18.01	0.89	12.21	0.39	15.44	0.68
21:0	1.09	0.27	0.93	0.16	0.99	0.20
22:0	16.04	6.92	13.48	4.12	14.87	5.42
23:0	1.01	0.83	0.87	0.62	0.95	0.72
24:0	12.66	15.89	12.88	12.56	12.69	14.22
25:0	1.49	0.92	1.67	1.82	1.53	0.93
26:0	15.40	27.76	20.60	28.12	17.02	27.83
27:0	1.75	2.90	2.35	2.93	1.92	2.95
28:0	12.14	18.72	17.13	20.31	13.78	19.59
29:0	2.11	4.53	2.76	4.90	2.22	4.84
30:0	6.79	14.66	9.62	16.61	7.65	15.81
31:0	0.34	0.96	0.44	1.17	0.35	1.08
32:0	0.60	4.12	0.74	6.14	0.65	5.20
33:0	0.04	—	0.05	—	0.02	—

due to the positional locations of fatty acids in the triacylglycerols of milkfat.

Experiments conducted on synthetic waxes showed that isomers with equal or close to equal chain lengths of alcohol and acid moieties had the highest melting points (Iyengar & Schlenk, 1969). Table 2 shows that the F32 fraction had C26:0 as the most abundant component in both acids and alcohols. Since the wax contents were not very different in the two fractions, the observed differences in the melting and crystallisation behaviour between the two fractions was mostly likely due to the small differences in the relative proportion of fatty acid constituent in these materials.

CONCLUSION

The results of this study demonstrated that slight differences in sediment composition may significantly affect its phase transition in oil. Thus, variety and environmental conditions may exert a significant influence on the sedimentation phenomenon in vegetable oils, including canola, as these factors are known to affect the relative proportions of the fatty acids and alcohols of waxy substances. Indeed, leaf waxes were reported to vary with variety and environmental conditions (Whitecross & Armstrong, 1972). It is not known to what extent such changes would influence the composition of canola wax constituent and therefore alter the propensity of canola oil to sediment formation after bottling.

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