EXAMINATION OF SEDIMENTS FROM CANOLA AND SUNFLOWER OILS.

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#### ABSTRACT

Sediments from Canadian and Australian canola oils were analyzed for composition and physical properties, and compared with sunflower oil sediment. Main component of sunflower sediment were waxes. Canola oil sediments comprised tri- and diglycerides, free fatty acids and alcohols and more than 20% of polar components. Both canola oil sediments contained 83% of saturated fatty acids with more than 24 carbon atoms, whereas sunflower sediment 50%. Long chain fatty alcohols in canola sediment made up 77% while in sunflower 48%. Canola oil sediments exhibited higher a melting point than sunflower sediment and this dictated the crystallization and formation of hazy material.

### INTRODUCTION

Precipitation of high-melting components causes a hazy appearance in vegetable oils such as sunflower (Chulu et al., 1989). Canola oil is known to contain a low level of these substances and normally does not require winterization. Occasionally, some batches of refined canola oil develop a hazy appearance on storage even at room temperature. Characterization of canola oil sediments revealed unique chemical and physical properties of this material (Hu et al., 1993; Liu et al., 1993; Gao and Ackman 1994). This paper provides comparison of physical and chemical properties of canola oil sediments from Canadian and Australian oils and sunflower oil sediment.

### EXPERIMENTAL

Sediments were isolated from industrial winterization cakes by extraction with hot benzene; chloroform mixture (5:1, v/v). Separated hulls from canola seeds were extracted similarly as winterization cakes. Obtained oils were stored at 0°C till sediments appeared then cold hexane was added in proportion 5:1 (v/v). Precipitation with fresh hexane was repeated three times. Sediments were filtered through teflon filter and finally washed with cold hexane. Composition of sediments was evaluated using TLC-FID (latroscan) for lipid classes and with GC-MS for fatty acid and alcohols composition and identification. Physical properties were analyzed by differential scanning calorimetry, X-ray diffraction and polarized-light microscopy.

# CHEMICAL COMPOSITION OF SEDIMENTS

TLC-FID evaluation of precipitates showed that main component in all sediments were waxes present at different proportion (Table 1).

| TABLE 1. | Composition | of oil sediments. |
|----------|-------------|-------------------|
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| Component           | Canadian | Australian | Sunflower | Hull |
|---------------------|----------|------------|-----------|------|
|                     | Canola   | Canola     |           |      |
| Waxes               | 72.3     | 74.2       | 99.4      | 48.4 |
| Triacylglycerides   | 1.3      | 2.1        | 0.0       | 22.6 |
| Diacylglycerides    | 2.7      | 2.4        | 0.0       | 0.5  |
| Free Fatty Acids    | 0.2      | 0.4        | 0.0       | 0.5  |
| Free Fatty Alcohols | 1.9      | 2.4        | 0.4       | 1.8  |
| Polar Compounds     | 21.3     | 22.8       | 0.2       | 26.4 |

Canola oil sediments were more complex than sunflower sediment, containing more than 20% of polar material which structure was not defined (Liu et al. 1995). Last components could not be moved on TLC with solvents used for phospholipids separation. Even application of more polar solvent mixture did not effect them. Further characterization showed that these components are not lipids because fatty acids presence was not detected neither react with phospholipids and glycolipids detection color reagents. Polar components carbohydrate origin has been suggested (Liu et al. 1995; Gao and Ackman 1995). Sunflower sediment contained only traces of polar component. Other components such as diglycerides and triglycerides were also detected which can initiate sediment formation when containing long chain saturated fatty acids (Liu et al., 1994; Gao and Ackman, 1995). Obviously, an efficient method is required to purify glycerides for their further characterization and understanding the role in sediment formation.

Detailed analysis of fatty acid and fatty alcohol composition of canola and sunflower waxes established presence of different proportions of long chain components (Table 2.). Most of the fatty acids and alcohols have even-number of carbon atoms. Canola oil sediments contained mostly saturated fatty acids with more than 22 carbon atoms. Whereas sunflower sediment was rich in fatty acids with 24 and fewer carbon atoms, this significantly differentiates those sediments. In total canola sediments contained 58% fatty acids with 26 and more carbon atoms while sunflower only 18%. Also presence of very long chain fatty acids, with 28 and more curbon atoms in the chain, was three times higher in canola than in sumflower sediments. A similar trend was observed in composition of fatty alcohols, where canola sediments contained 83% of long chain compounds, while sunflower only 48%. As expected, differences in composition directly affected physical properties of sediments. DSC curves showed higher melting temperature for canola oil sediments than for sunflower. For both types of sediments X-ray diffraction showed similar patterns, typical for waxes (Liu et al., 1993). A polarized light microscope revealed that both sediments often crystallized as elongated rods. Domination of both long chain alcohols and fatty acids in canola sediments can be detrimental factor in formation of sediment in refined oil.

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TABLE 2. Fatty acids and alcohols composition of canola and sunflower sediments.

| COMP.        | CANADIAN<br>CANOLA |                  | AUSTRALIAN<br>CANOLA |       | SUNFLOWER |       | HUI   | HULL  |  |
|--------------|--------------------|------------------|----------------------|-------|-----------|-------|-------|-------|--|
|              | FA'                | FAL <sup>b</sup> | FA                   | FAL   | FA        | FAL   | FA    | FAL   |  |
| 12:0         | 0.10               |                  | 0.13                 |       | 0.10      |       | -     | 0.59  |  |
| 14:0         | 0.37               |                  | 0.26                 |       | 0.19      |       | 0.41  | 2.46  |  |
| 15:0         | 0.09               |                  | 0.06                 |       | 0,11      |       | 0.12  | ~     |  |
| 16:0         | 2.10               |                  | 2.23                 |       | 4.23      |       | 2.83  | 1.01  |  |
| 16:1         | 0.32               |                  | 0.30                 |       | 0.26      |       | 0.21  |       |  |
| 17:0         | 0.21               |                  | 0.28                 |       | 0.11      |       | 0.20  | 0.33  |  |
| 18:0         | 2.86               | 0.05             | 2.77                 | 0.06  | 5.95      | 0.90  | 2.69  | 1.17  |  |
| 19:0         | 0.20               |                  | 0.13                 |       | 0.35      | 0.01  | 0.20  | 0.06  |  |
| 20:0         | 9.38               | 0.25             | 9.40                 | 0.38  | 38.49     | 1.85  | 9.19  | 2.75  |  |
| 20:1         | 0.46               |                  | 0.21                 |       | 0.12      |       | 0.26  |       |  |
| 21:0         | 0.68               | 0.11             | 0.60                 | 0.10  | 0.64      | 0.19  | 0.73  | 0.39  |  |
| 22:0         | 9.42               | 3.48             | 9.19                 | 3.63  | 20.70     | 13.81 | 10.34 | 7.29  |  |
| 22:1         | 0.25               |                  | 0.19                 |       |           |       | 0.24  |       |  |
| 23:0         | 0.58               | 0.54             | 0.48                 | 0.55  | 0.18      | 1.14  | 0.52  | 0.29  |  |
| 24:0         | 13.26              | 10.97            | 13.95                | 11.26 | 10.44     | 31.28 | 14.07 | 5.76  |  |
| 24:1         | 0.33               |                  | 0.41                 |       | 0.12      |       | 0.36  |       |  |
| 25:0         | 0.92               | 0.18             | 0.96                 | 80.0  | 0.27      | 2.28  | 0.76  | 0.75  |  |
| 25:1         | 0.36               |                  | 0.42                 |       | 0.01      |       | 0.43  |       |  |
| 26:0         | 26.13              | 25.68            | 25.62                | 26.29 | 6.51      | 23.53 | 23.34 | 20.35 |  |
| 26:1         | 0.18               |                  | 0.21                 |       | 0.02      |       | 0.31  | _     |  |
| 27:0         | 2.02               | 2.68             | 2.03                 | 1.96  | 0.39      | 0.62  | 2.21  | 0.51  |  |
| 28:0         | 16.92              | 20.76            | 16.72                | 21.82 | 7.18      | 11.48 | 16.76 | 19.30 |  |
| <b>28:</b> 1 | 0.22               |                  | 0.31                 |       | 0.03      |       | 0.29  |       |  |
| 29:0         | 2.47               | <b>5.</b> 29     | 2.08                 | 4.98  | 0.41      | 0.53  | 2.56  | 0.55  |  |
| 30:0         | 8.26               | 19.83            | 9.08                 | 19.76 | 2.98      | 6.11  | 8.79  | 26.78 |  |
| 31:0         | 0.06               | 1.39             | 80.0                 | 1.33  | 0.12      | 0.36  | 0.08  | 0.76  |  |
| 32:0         | 1.27               | 7.35             | 1.30                 | 7.38  | 0.06      | 5.27  | 1.49  | 6.11  |  |
| 33:0         | 0.36               | 0.38             | 0.35                 | 0.42  | 0.02      | 0.25  | 0.37  | 0.74  |  |
| 34:0         | 0.22               |                  | 0.25                 |       | 0.01      |       | 0.24  |       |  |

<sup>\* -</sup> Fatty acid; \*- Fatty alcohol

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