

# Hybrid hazelnut oil characteristics and its potential oleochemical application<sup>1</sup>

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## Abstract

The demand for vegetable oils as alternatives to petroleum is growing. Vegetable oils offer performance advantages and are environmentally friendly. Soybean oil is one of the promising resources and has been studied extensively. However, soybean oil lacks thermal and oxidative stability and has undesirable cold flow properties. These disadvantages are directly related to its fatty acid composition. Therefore, new varieties with superior oil traits are needed. Hybrid hazelnut oil is just such an attractive raw material. The objective of this study was to investigate hybrid hazelnut oil fatty acid composition and several other important properties in view of potential oleochemical applications. Hazelnut oil, derived from hybrid hazelnuts (*Corylus sp.* L.) grown in southeastern Nebraska, was extracted using a Soxhlet extractor. The oil contents of 25 different hybrid hazelnut samples ranged from 51.4% to 75.1% of the dry weight of nut kernels. Fatty acid profiles were analyzed by gas chromatography. It was found that oleic and linoleic acids comprised more than 90% of fatty acids in the oil. Oxidative onset temperature (OOT) was used as an indicator of thermal stability. The OOT of the hazelnut oils were in the range of 184.7 to 190.4°C, which were significantly higher than that of soybean oil (148.4°C). The cloud points of hazelnut oils (-12.4 to -14.9°C) were lower than that of soybean oil (-9.9°C). The iodine values were in the range of 90.6 to 97.4. The kinematic viscosity of d

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<sup>1</sup> A contribution of the University of Nebraska Agricultural Research Division, supported in part by funds provided through Hatch Act.

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hazelnut oil was  $65.7 \pm 2.2 \text{ mm}^2/\text{s}$  at 22 °C. These initial analyses suggest that hazelnut oil is a potential feedstock for oleochemicals.

*Key words:* Hybrid hazelnut; Hazelnut oil; Fatty acid composition; Iodine value; Thermal stability; Cloud point; Kinematic viscosity.

## **1. Introduction**

High crude oil prices and concern about environmental pollution caused by limited and nonrenewable petroleum resources have encouraged scientists to develop and utilize renewable energy resources to diversify our energy supply. Vegetable oils are ideal alternatives to petroleum oil products because they are renewable and environmentally friendly (e.g. readily biodegradable, carbon neutral and low in toxicity). Furthermore, vegetable oil-based products hold great potential for stimulating rural economic development because farmers would benefit from increased demand for vegetable oils. Different vegetable oils, including palm oil, soybean oil, sunflower oil, rapeseed oil, and canola oil have been used to produce biodiesel fuel and lubricants (Kalam and Masjuki, 2002; Bondioli, 2003; Demirbaş, 2003). With petroleum prices rising continually, the demand for vegetable oil based products is expected to climb considerably (USDA, 2005). Of the vegetable oils, soybean oil ranks first in worldwide production (29%), and represents a tremendous renewable resource (Rose and Rivera, 1998). However, one of the roadblocks to the market appeal of soybean oil-based products is its lack of thermal and oxidative stability and high pour point. The instability of oil is related directly to the unsaturated bonds in the residual fatty acids (Anonymity, 2003). Further, the degree of unsaturation influences the reactivity of the double bonds. Double bonds in polyunsaturated systems are more reactive than monounsaturated double bonds (AOCS, 2003). Soybean oil has the most

challenging profile since it contains a relatively high percentage of linoleic acid (18:2, 52%) and linolenic acid (18:3, 7%) and a low percentage of oleic acid (18:1, 25%) (Table1). Many efforts have been made to chemically modify the linoleic and linolenic acids, which are known to have the poorest oxidation stability (Hwang and Erhan, 2001; Huang et al., 2003; Vicray et al., 2004). DuPont also has used biotechnology to develop genetically modified high oleic acid (83%) soybeans. Although genetically modified soybean oil is more stable than the conventional oil which contains only 20% oleic acid, technical and economic barriers and public acceptance of genetically modified materials limit its application. Therefore, new varieties with superior oil traits are needed. Hazelnut oil is just such an attractive raw material.

Hazelnut (*Corylus sp. L.*), a member of the birch family, Betulaceae, is an important commercial and subsistence crop in many countries including Turkey, Spain, Italy and USA (Bagley and Sutton, 2002; Ercisli and Read 2001). Hazelnuts have been cultivated and selected from wild populations of the European hybrid hazelnut (*Corylus avellana*) in Europe over many centuries. In the USA, the vast majority of commercial hazelnut production comes from the Willamette Valley in Oregon (Mehlenbacher, 2003), where on average, 25,000 metric tons are produced each year (Chase and Anagnostakis, 2003). These orchards are planted to cultivars of the European hazelnut (*Corylus avellana* L) (Farris 2000). Since the early 1900s, breeders in the United States have attempted to develop commercial-quality hybrid hazelnut shrub cultivars with cold hardiness and resistance to Eastern Filbert Blight (*Anisogramma anomala* Peck) through interspecific crosses between the European hazelnut, *Corylus avellana*, and the North American natives, *Corylus americana* and *C. colurna* L. (Weschcke, 1954; Slate, 1961; as cited by Molnar, 2004).

In recent years, much interest has been generated for the use of the hybrid hazelnut as an alternative crop in the upper midwestern region of the USA (Ercisli and Read, 2001). A nine-acre hybrid hazelnut planting at Arbor Day Farm, Nebraska City, NE, established in 1996, currently is one of the largest plantings of hybrid hazelnuts in the Central States region. The planting consists of 5,200 genetically distinct hybrid hazelnut shrubs grown from open pollinated seedlings derived from a plantation breeding program at Badgersett Research Corporation, Canton, MN. Recent research conducted with the Arbor Day Farm hybrids indicates that the three-year average of the highest yielding selections produce an equivalent of 4 tonnes/ha of husked nut (Hammond, 2006). These plants are not cultivated intensively, receive little weed control except mowing between rows, are planted much too close together (spaced < 2m within the row by < 5m between rows), and are not fertilized or irrigated (Figure 1). Under more intensive cultivation, yields are expected to be higher. Initial tests also indicate that 60% of the top 10 cultivars showed no symptoms of Eastern Filbert Blight one year after intensive exposure to the disease. Nuts produced, while numerous, are generally small, averaging 1.2 - 2.4 g/husked nut, and are 30 - 46% kernel by weight, depending on the selection (Figure 2).

Currently, hazelnut oil is used mainly in salad dressings and cosmetic and pharmaceutical products. There is little information on industrial applications of hazelnut oil. The objective of this research project was to investigate hybrid hazelnut oil fatty acid composition and to screen several other important properties in view of potential oleochemical applications.

## **2. Material and method**

### *2.1 Materials*

Hybrid hazelnuts, selected from high yielding shrubs, were hand harvested from Arbor Day Farm, Nebraska City, NE. Husks (or the involucre) were removed mechanically and nuts were sorted mechanically by size and cracked using mechanical crackers. Sample of ten nuts were selected randomly from each of twenty-five plants (top 25 producers) to measure the crude oil contents. Further, six oils were selected for fatty acid profile, iodine value, oxidative onset temperature, cloud point, and kinematic viscosity analyses. Soybean oil was purchased from a supermarket and used as the control. Hexane, boron trifluoride/methanol reagent, iodine /chlorine reagent, sodium thiosulfate and other reagents were of reagent grade, and were purchased from Fisher Scientific (Pittsburgh, PA).

## *2.2. Oil content*

The oil contents of hazelnuts were determined according to AACC Method 30-25 (1991). The nuts from different shrubs were first ground separately and dried thoroughly in a vacuum oven (80 °C). The dried ground samples (approximately 3 g) were placed in a thimble and the oil was extracted using 50 ml of hexane in a Soxhlet extractor for 8 h. After the extraction, the hexane was evaporated and the collection cups containing the extracted oil were placed in a vacuum oven at 95 °C for 1 h to remove all traces of hexane. The cups were cooled and weighed. The oil content was expressed as the following:

$$\% \text{ oil content} = \frac{\text{Weight of oil}}{\text{Weight of sample}} \times 100$$

## *2.3. Fatty acid profile*

The fat acid profiles of hazelnut oils were determined by gas chromatography. The oils (2 drops of each) first were saponified into individual acids. The fatty acids were then converted

to volatile methyl ester derivatives by reacting with boron trifluoride/methanol reagent (2ml) in a heater for 30 min. Hexane (1 ml) and water (2 ml) were added to the samples. The samples were centrifuged and the methyl ester derivatives of the fatty acids were dispersed in hexane. The hexane layer was separated and 1 µl was injected into the chromatograph using a microliter syringe. An HP 5890 Series II Gas Chromatograph (Hewlett Packard Company, Wilmington, DE) equipped with a flame ionization detector and a Hewlett Packard 3396 A Integrator (Avondale, PA) was employed to obtain chromatograms. Helium was the carrier gas with a head pressure of 20 psi. A fused silica capillary column DB-Wax (30 m x 0.25 mm i.d.) with a 0.25 µm coating (Alltech, Deerfield, IL) was used as the stationary phase. Injector, detector and oven temperatures were 250, 250, and 200 °C, respectively. Peaks were identified by comparing their retention times with a fatty acid methyl esters standard. The soybean oil was treated in the same way and tested using the same procedure

#### 2.4. Iodine value

Iodine values of the hazelnut oils were measured using ASTM D 1959-97 method (1997). An iodine /chlorine reagent (Wijs solution, 25ml) was added to react with the double bonds in the oil. The amount of Wijs solution absorbed was determined by back titrating the excess Wijs solution using sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ) and comparing it to a blank sample.

$$\text{Iodine value (I)} = \frac{(B - V) \times N \times 12.69}{S}$$

where V is the amount of  $\text{Na}_2\text{S}_2\text{O}_3$  solution required for titration of the specimen, (mL); B is the amount of  $\text{Na}_2\text{S}_2\text{O}_3$  solution required for titration of the blank,(mL); N is normality of the  $\text{Na}_2\text{S}_2\text{O}_3$  solution; and S is the sample used (g).

### *2.5. Oxidative onset temperature*

Oxidation onset temperatures (OOT) were determined by differential scanning calorimetry (DSC, Mettler, Columbus, OH, USA) using ASTM E 2009-02 method (2002). The test specimens (about 3 mg), in an aluminum pan, and an empty reference aluminum pan were heated at a heating rate of 10 °C/min in an air environment. Heat flow out of the specimen was monitored as a function of temperature until the oxidative reaction was manifested by heat evolution on the thermal curve. OOT is defined as the temperature when a rapid increase in the rate of oil oxidation is observed. This temperature was obtained by extrapolating the tangent drawn on the steepest slope of the reaction exothermic curve.

### *2.6. Cloud point*

Cloud points of the oils were determined using DSC (Mettler, Columbus, OH, USA). The oil samples (35-40 mg) were cooled from 10 °C to -40 °C at a cooling rate of 1 °C/min, staying at -40 °C for 2 min, and then heated to 10 °C at a heating rate of 1 °C/min. The cloud point was the onset temperature of the initial small exothermic peak on the cooling curve.

### *2.7. Kinematic viscosity*

The dynamic viscosity of the hazelnut oils and soybean oil were determined with a Viscotester VT500 (Haake, Mess-Technik, Germany) with sensor system of cone and plate PK1-0.5 deg at 22 °C. The sample (100 uL) was pipetted onto the plate. The cone was rotated at 500 rpm, until the reading stabilized (about 2 min). Five readings were taken at intervals of 40-60 s. The viscosity tests were performed three times to obtain the mean dynamic viscosity. Kinematic viscosity was calculated by dividing dynamic viscosity by the oil density.

### **3. Results and discussion**

#### *3.1. Oil content*

Hazelnuts are particularly valuable for their lipids. Oil contents of the top 25 producing plants in the planting (Arbor Day Farm, Nebraska City, NE) ranged from 51.4% to 75.1% of the weight of the kernel. Combining the nut yield, percent kernel and oil content of the kernel indicate production rates of approximate 1000 kg of oil per ha (3m x 5m spacing, 725 shrubs/ha), which compared very favorably with soybean yields (~500 kg/ha). Soybean has an oil content of approximately 20% on a dry basis. Although soybeans are the second most valuable crop in the United States, behind corn, the main product from soybeans is protein (~40% dry basis), with oil being a by-product of processing the protein. The high oil content of hazelnuts makes them worthy of note with respect to their potential commercial value. It also is worthy of note that industrial solvent extractions will result in a slightly lower yield (< 1%) than solvent extraction in a Soxhlet.

#### *3.2. Fatty acid profile*

Oleic, linoleic, and linolenic acids are the major fatty acids found in oil seeds, and the ratio of these three fatty acids differs in different oil seeds. Gas chromatograms of a typical (hybrid hazelnut oil and soybean oil are presented in Fig.3, and fatty acid compositions of hazelnut and soybean oils are summarized in Table 1. The hazelnut oil contained high amounts of oleic (18:1, 68.8%- 78.6%) and linoleic (18:2, 14.2%-23.3%) acids, and low amounts of palmitic (16:0, 4.5%-5.9%), stearic (18:0, 0.5% - 2.8%), and linolenic (18:3, 0.1-0.2%) acids. Oleic acid was the predominant fatty acid, and oleic acid and linoleic acid comprised more than 90% of the fatty acid composition in all nuts. The hazelnut oil fatty acid profiles were not

significantly different between samples (shrubs). On the other hand, the soybean oil contained high amounts of saturated (palmitic (11.4%) and stearic (4.2%)) acids and polyunsaturated (linoleic (52.1%) and linolenic (7.6%)) acids, and a moderate amount of monounsaturated oleic acid (24.7%). The important acids for thermal and oxidative stability are those labeled xx:2 and xx:3, which indicate the number of active sites on each chain. The more sites, the less stable the chain. The acids that most significantly affect for freezing point are those labeled xx:0 (United Soybean Board, 2004). Oils containing fully saturated acids tend to solidify in cold weather, limiting their low-temperature functionalities. From this standpoint, soybean oil has a challenging profile. Chemical or genetic modifications have been conducted to increase oleic acid content or to remove the linolenic materials. Compared to soybean oil, the unique composition (intrinsic high oleic acid content and lower levels of saturates) of hazelnut oil should facilitate its use in many different oleochemical applications.

### *3.3. Iodine value*

The iodine value is a measure of the unsaturation of oils and is expressed as the number of centigrams of iodine per gram of sample. The iodine values of the hazelnut oils ranged from 90.6 to 97.4, which were much lower than that of soybean oil (130.4) (Table 1). The differences in iodine values between soybean and hazelnut oils were due to the different fatty acid compositions in the two types of oil molecules. Soybean oil has high a polyunsaturated fatty acids content, whereas hazelnut oil contains mostly monounsaturated fatty acids. Iodine value is not the best index for oil stability since it does not take into account the positions of the double bonds available for oxidation. However, it still is important in assessing the stability of an oil in oleochemical applications, e.g. biodiesel fuel standards specify an upper limit. For example,

Europe's EN 14214 specification allows a maximum iodine value of 120 and Germany's DIN 51606 tops out at 115. The European and German specifications result in a defacto ban on soybean based biodiesel (Wherley, 2005).

#### *3.4. Oxidative onset temperature*

Oxidative stability affects biodiesel by contact with air during storage and affects lubricants through contact of the lubricant with hot engine parts, accelerating the oxidation process (Knothe and Dunn, 2003). Some methods, including oil stability index and bis-allylic/allylic position equivalent (APE/BAPE), were employed to determine oil stability (Knothe, 2002). In our experiment, oxidative onset temperature (OOT) was used to evaluate the oxidative stability of the oils. A higher OOT value indicated a more stable material. The DSC curves of typical hazelnut and soybean oils are shown in Fig.4, and OOTs of hazelnut and soybean oils are summarized in Table 1. Hazelnut oil had an OOT of 184.7-190.4 °C, while the OOT of soybean oil was 148.4 °C. An approximate 40 °C higher OOT of hazelnut oil than its soybean counterpart can be explained by the corresponding fatty acid compositions. The degree of unsaturation influences the reactivity of the double bonds. Double bonds in polyunsaturated systems are more reactive than monounsaturated double bonds (AOCS, 2003). The high oleic acid content in hazelnut oil increases its thermo oxidative stability, while the linoleic and linolenic acids in soybean oil are vulnerable to attack by oxygen.

#### *3.5. Cloud point*

The cloud point is the temperature at which a cloud is formed in an oil caused by the first stage of crystallization. It is of importance since it limits the flow properties, which influence the

use of oil in a cold environment. The cloud points of hazelnut and soybean oils were determined by DSC, and the cooling and heating curves of a typical hazelnut oil and soybean oil are shown in Fig. 5. The cooling curve of hazelnut oil showed an initial very small exothermic peak at -12.4 °C. This peak corresponded to the first stage of crystallization and cloud point corresponded to the onset of crystallization. A large exothermic peak at -31.5 °C with further cooling indicated the oil underwent a main crystallization step. The oil had a melting peak at -6.1 °C. For soybean oil, the initial crystallization occurred at -9.9 °C, and no major crystallization was observed. Soybean oil had a melting peak at -15.9 °C. The lower cloud point of hazelnut oil was a result of the lower content of saturated (palmitic and stearic) acids.

### *3.6. Kinematic viscosity*

Soybean oil had a kinematic viscosity of  $58.9 \pm 3.3 \text{ mm}^2/\text{s}$ , while the kinematic viscosity of hazelnut oil was  $65.7 \pm 2.2 \text{ mm}^2/\text{s}$  at 22 °C (Table 1). The slightly greater viscosity of hazelnut oil was attributed to the different fatty acid composition. Soybean oil has a high percentage of polyunsaturated acids, while monounsaturated acid was the major acid in hybrid hazelnut oil. Double bonds reduce kinematic viscosity in fatty compounds (Knothe and Steidley, 2005). The viscosity of hazelnut and soybean oils must be reduced for biodiesel application since the kinematic viscosity of biodiesel fuel standard in the United States is 1.9-6.0  $\text{mm}^2/\text{s}$ , and the high viscosity often results in operational problems such as engine deposits (Knothe and Steidley, 2005).

### *3.7. Potential oleochemical application*

Based on the above several important evaluations, hazelnut oil is a promising feedstock for oleochemical applications. These potential applications include (1) biodiesel fuel which typically is produced by transesterification reaction to replace the glycerol molecules with methanol; (2) biolubricants which can be prepared by slight chemical modification (hydroxylation and esterification reactions); and (3) biopolymers, e.g. polyurethane which can be manufactured by hydroxylation and polymerization reactions. Preparation and characterization of these oleochemicals will be investigated in separate studies.

#### **4. Conclusions**

The oil contents of hazelnuts were in the range of 51.4% to 75.1% of dried weight of the kernel. Combining the nut yield, percent kernel and oil content of the kernel, the oil yield of hazelnuts was approximate 1000 kg per ha, about 2 times that of soybeans. The hazelnut oils were unique in that only two fatty (oleic and linoleic) acids accounted for 90% of their fatty acid content. High oleic and linoleic acids contents and low linolenic acid content improved the oils thermo oxidative stability, while low levels of saturated (palmitic and stearic) acids enhanced their properties in cold environments. The iodine values of 90.6 to 97.4 meet the specification for biodiesel fuel. Hybrid hazelnut oil had a kinematic viscosity of  $65.7 \pm 2.2 \text{ mm}^2/\text{s}$ . These properties suggest that hazelnut oil is a potential source for oleochemical.

## **Acknowledgements**

The authors are grateful to Dr. Susan L. Cuppett, Food Science & Technology Department, for use of her GC and for her guidance in GC operation and chromatogram analyses. We appreciate Elizabeth Hammond, School of Natural Resource, providing some data. We also express our gratitude to Dr. Dennis Wiesenborn at North Dakota State University and Dr. Bernie Tao at Purdue University for measuring viscosities.

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Table 1

Characteristics of several hybrid hazelnuts and soybean oils

Characteristics of the oil	Value	
	Soybean oil	Hybrid hazelnut oil
Crude oil fat (%)	~20 <sup>a</sup>	51.4-75.1
Fatty acid composition (%)		
C16:0	11.4	4.5-5.9
C18:0	4.2	0.5-2.8
C18:1	24.7	68.8-78.6
C18:2	52.1	14.2-23.3
C18:3	7.6	0.1-0.2
Iodine value (g iodine absorbed /100g sample)	130.4	90.6-97.4
Oxidative onset temperature (° C)	148.4	184.7-190.4
Cloud point (° C)	-9.9	-12.4-- -14.9
Kinematic viscosity at 22 ° C (mm <sup>2</sup> /s)	58.9 ± 3.3	65.7 ± 2.2

<sup>a</sup> From Wikipedia, the free encyclopedia

Figures caption

Figure 1. Hybrid hazelnut shrubs

Figure 2. Hybrid hazelnuts

Figure 3. Gas chromatograms of soybean oil and hazelnut oil

Figure 4. The DSC curves for oxidative onset temperature of hazelnut and soybean oils

Figure 5. The DSC cooling and heating curves for cloud point of hazelnut and soybean oils



Figure 1. Xu et al.



Figure 2. Xu et al.

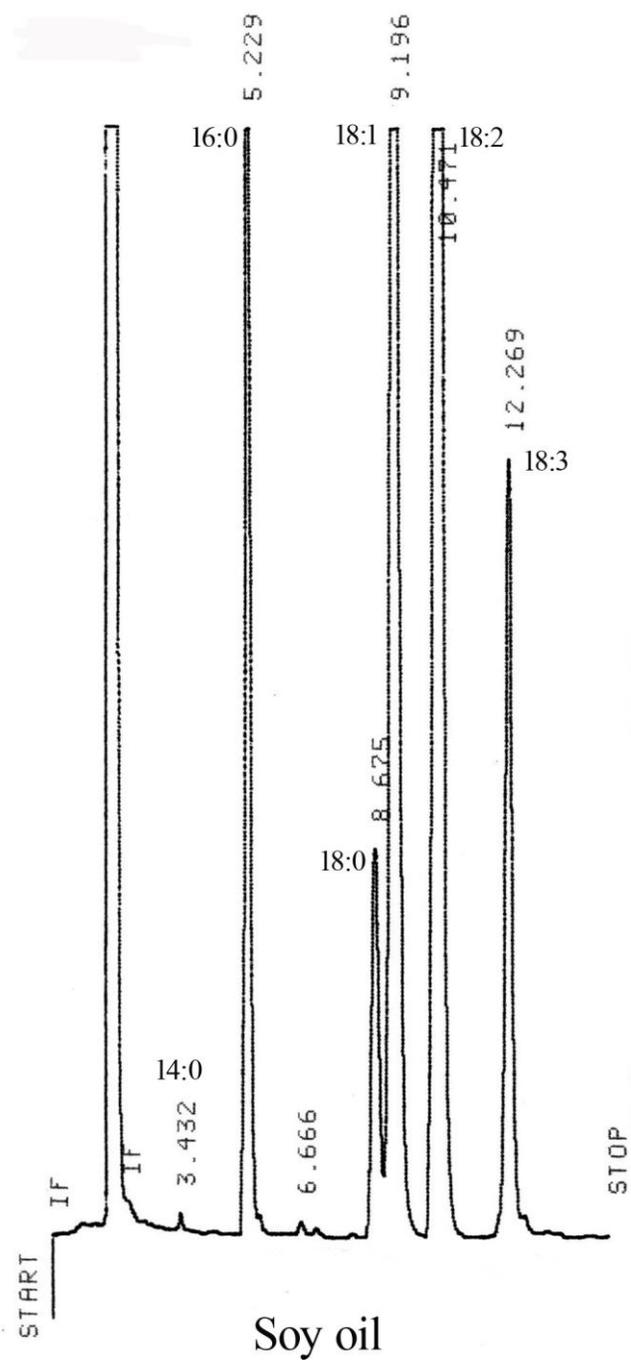


Fig.3. Xu et al. (continued)

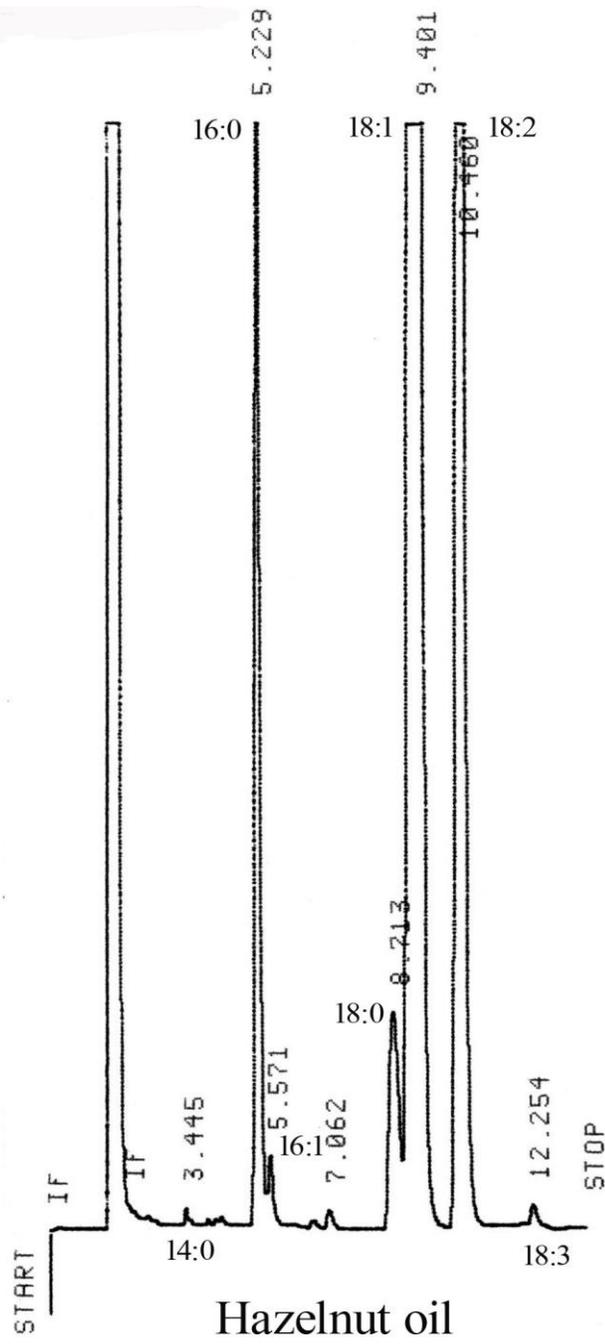


Fig.3. Xu et al.

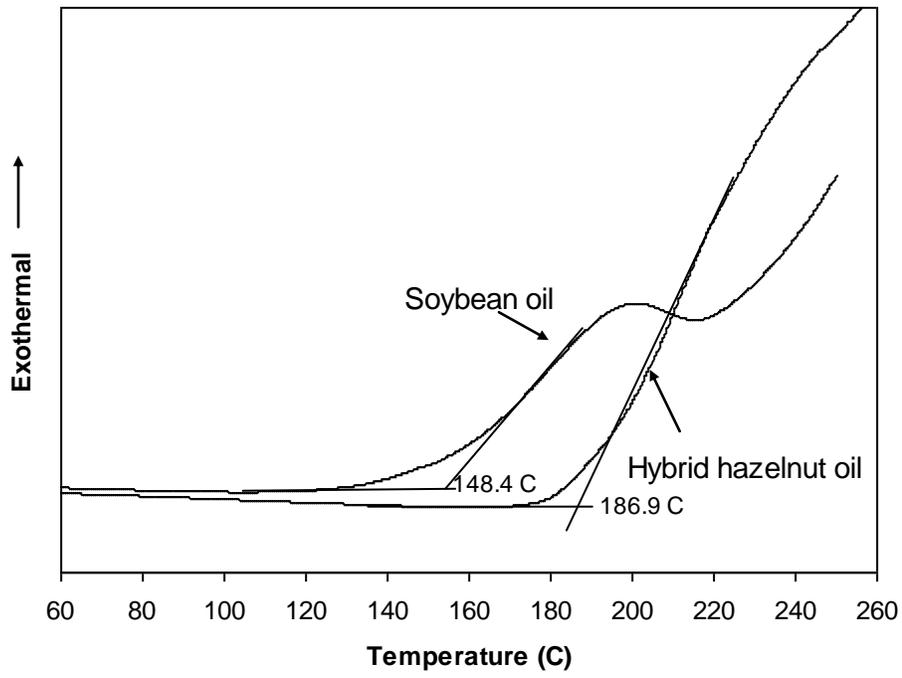


Fig.4 Xu et al.

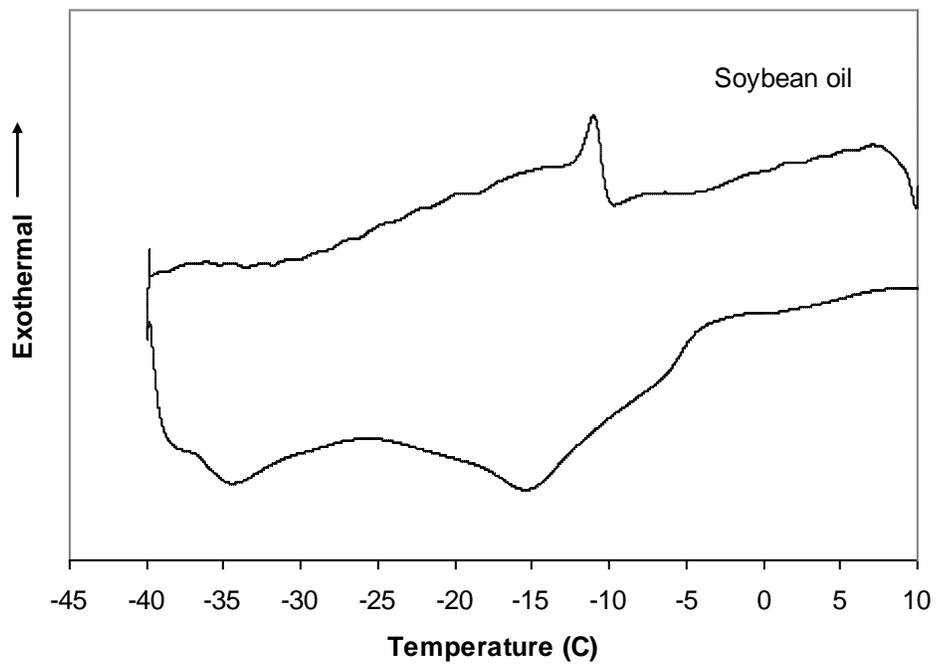
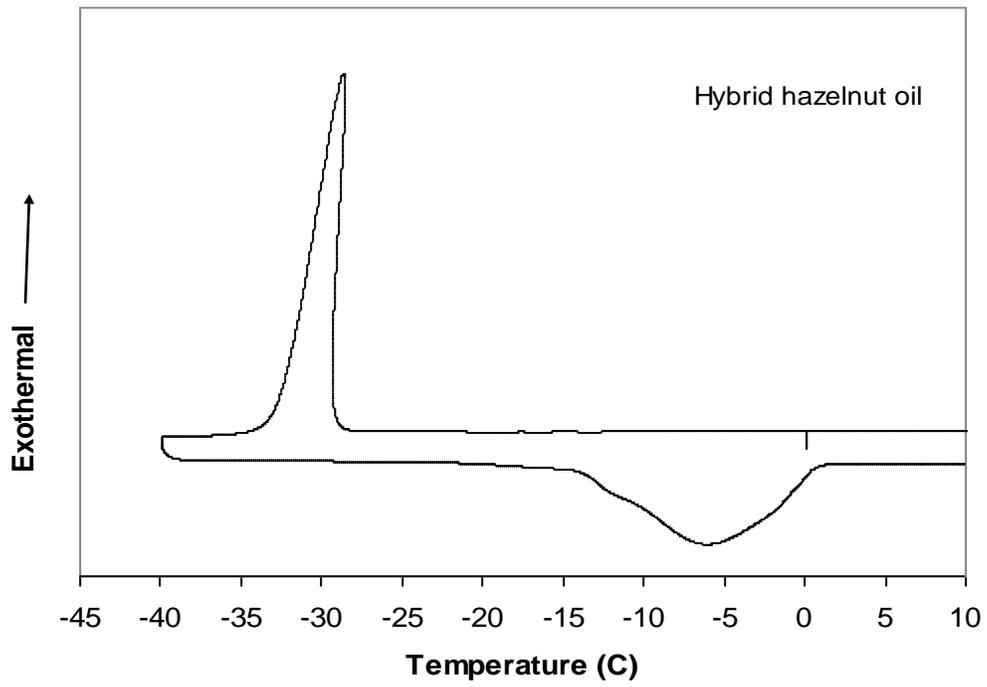


Fig. 5. Xu et al.