Heat Transfer Properties of a Series of Oxidized and Unoxidized Vegetable Oils in Comparison with Petroleum Oil-Based Quenchants

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Vegetable oils, especially soybean oil, exhibit substantially poorer thermal-oxidative stability than commercially available petroleum oil quenchant formulations. Therefore, to achieve any commercially interesting performance, vegetable oils must be stabilized by the addition of antioxidant inhibitors. This work describes the ability of two commercially available antioxidants, Irganox L 57 and Irganox L 109, to stabilize soybean oil against thermal-oxidative degradation. In addition, the effect of antioxidant stabilization on quenching performance was evaluated by determining the profile of heat transfer coefficient variation throughout the quenching process at different times after being subjected to an accelerated thermal-oxidation aging test. The results of this work are discussed here.

1. Introduction

Properties, such as hardness and strength, of carbon and alloy steels are dependent on the cooling time-temperature profile (cooling curve) exhibited by the quenching medium relative to the TTT (time-temperature-transformation) or CCT (continuous cooling transformation) curves for the steel of interest. Quenchants not only control properties but also, depending on the quenching process and quenchant selection, control the residual stress profile with the steel and are critically important to optimize distortion control and prevent cracking. Depending on the steel and hardening process, the most common quenchants include air, oil, water, brine, aqueous polymer solutions, and high-pressure gas quenching.

Of the vaporizable quenchants, petroleum oil-derived fluids continue to be the most commonly encountered ones throughout the industry globally. However, there is an ongoing effort to replace petroleum oil-derived industrial fluids because of the price and the environmental and toxicological property hazards exhibited by petroleum products. Various quenching media have been evaluated as alternatives to petroleum oil including water, especially utilizing water in conjunction with time quenching (Ref 1-3) or by intensive quenching (Ref 4), water-gas (Ref 5), aqueous polymers (Ref 6, 7), and high-pressure gas (Ref 8). However, petroleum oil-derived quenchants continue to dominate the marketplace.

Currently, there is increasing interest in the use of bio-based oils as replacements for petroleum oil base stocks for lubricant and quench oil formulation. Bio-based oils are defined as "industrial products including fuels, but not food or feed, made from renewable agricultural or forestry resources." Seed oils are currently one of the most common sources of bio-based oils and among the various seeds from which seed oils are derived. Soybeans are in the greatest production worldwide and the United States has the highest soybean production rate followed by Brazil (Ref 9). Of the various seed oils produced in the USA in 2004, soybean oil has the largest production rate and is therefore one of the oils of the greatest interest for use in replacing petroleum oil-derived quenchants (Ref 10).

Tagaya and Tamura compared the quench severity of different vegetable oils including soybean, rapeseed, and castor oils with mineral oils and fish/animal oils with respect to fluid source and viscosity and oxidative stability for various naturally derived fluids (Ref 11). Fujimura and Sato followed this work in 1963 by examining the quenching performance of the vegetable oils reported by Tagaya and Tamura and different petroleum oil quenchants and concluded that the performance of soybean and rapeseed oil was essentially equivalent (Ref 12).

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Currently, the most commonly cited vegetable oil base stocks used for quenchant formulation are canola oil (Ref 13) and soybean oil derivatives (Ref 14). Although vegetable oils exhibit a cooling performance different from the petroleum oil standard used, in none of the references was any systematic comparison of vegetable oil structure with oxidative stability and its corresponding impact on quenching performance demonstrated. The cooling curves reported were obtained according to the ISO 9950 (Ref 15), which is essentially equivalent to the ASTM D6200 "Standard Test Method for Determination of Cooling Characteristics of Quench Oils by Cooling Curve Analysis" (Ref 16). Both the ISO 9950 and ASTM D6200 utilize a 12.5-mm-dia × 60-mm INCONEL 600 cylindrical probe with a Type K thermocouple inserted into the geometric center.

Oxidative stability performance improvements of vegetable oils can be achieved by chemical or genetic modifications (Ref 17-19) or by process improvements such as winterization and partial hydrogenation (Ref 20, 21). Winterization (fractionation) is performed to remove crystallized fats and improve the pour point of the base oil. The performance objective is to reduce the linolenic and linoleic ester content of the vegetable oil to increase the oxidative stability, making the resulting vegetable oil more suitable for use in industrial applications (Ref 20). Honary demonstrated that the oxidative stability of soybean oil is substantially improved by partial hydrogenation. However, this also increases the level of saturation in the oil, which produces crystallized fats which may be removed by a winterization process (Ref 21).

Genetic engineering has produced soybean oil with >85% oleic acid ester contents (Ref 17, 18). There are, however, a number of substantial problems with genetic engineering to reduce polyunsaturation including costs associated with regulatory approval.

Totten et al. reported the results of cooling curve, hardenability, heat transfer, and wettability characterization studies conducted with crude and partially hydrogenated and winterized soybean oils provided by Honary (Ref 21, 22). The soybean oils investigated showed no significant differences in cooling behavior and the rewetting conditions on the test specimen surface, and the cooling rates were similar. Comparison of the cooling time-temperature and cooling rate curves showed that the vegetable oils exhibited faster cooling rates than the mineral oil used as a reference (Ref 22).

Although there are various references to the quenching performance of soybean oil and other vegetable oils such as the work reported by Prabhu (Ref 23, 24), with the exception of Tagaya and Tamura (Ref 25) and Canale et al. (Ref 26), no one has reported the effect of the oxidative degradation on quenching performance and the ability to stabilize cooling performance, as determined by cooling curve performance with the addition of antioxidants. The objective of this work was to study the degradation of quenching performance of soybean oil with and without the addition of antioxidants.

2. Experimental Methods and Materials

The soybean oil used for this work was purchased at the local market in Sao Carlos, Brazil, and was used in the

Table 1 Composition of soybean oil/antioxidant blends and their designation codes

| Concentration of antioxidant	Name used
Soybean—as purchased	SO
SO + 0.3% Irganox L 57 + 0.7% Irganox L 109	SO1
SO + 1% Irganox L 109	SO2
SO + 1% Irganox L 57	SO3
SO + 0.5% Irganox L 57 + 0.5% Irganox L 109	SO4

"as-purchased" condition.* Quenching performance of these oils was compared to two commercially available petroleum quenching oils: MicroTemp 157—a conventional "slow" oil; MicroTemp 153B—an accelerated "fast" oil. The two antioxidants used for this work are Irganox L57 and Irganox L109. Both were used as supplied by Ciba Geigy Corporation. The composition of the soybean oil blends prepared for this study is shown in Table 1. Viscosity was measured at 40 and 100 °C according to ABNT NBR 10441—10/02 (Ref 28). The oxidative stability of the vegetable oils and quenching oil was determined using an accelerated oxidation test previously reported by Bashford and Mills (Ref 29) and with equipment built for this purpose by Farah** (Ref 30). A schematic illustration is provided in Fig. 1. The oxidation test was conducted as follows: Into a 2300-mL glass vessel (12.5 cm diameter, 19 cm in height) was added 2000 mL of the vegetable oil which was heated to 150 ± 2 °C for 15 min with agitation. During this time, the fluid was aerated using a gas sparge tube at 4 L air/h. (The agitation was provided by an electrically driven propeller mixer with speed settings of 0-10, and a setting of 6 was used.) After 12 min, the electrical resistance immersion heater, aeration system, and agitation were automatically shut off for 15 min during which time the fluid was cooled to 125 ± 2 °C in 3 min. The fluid was reheated again with agitation and air sparging and these 15-min cycles were repeated over the test duration of 48 h. Cooling curves were obtained under unagitated conditions according to ASTM D6200-01—"Standard Test Method for Determination of Cooling Characteristics of Quench Oils by Cooling Curve Analysis"—at a bath temperature of 40 °C. This test method is based on the 12.5-mm-dia. × 60-mm cylindrical INCONEL 600 probe assembly. After heating the probe in a furnace to 850 °C (1562 °F), it was manually and rapidly immersed into 2000 mL of the oil to be tested which was contained in a tall-form stainless steel beaker. The probe temperature and cooling times are recorded at selected time intervals to establish a cooling temperature versus time curve.

3. Calculation of Heat Transfer Coefficients

Heat transfer processes are complex and the heat transfer coefficient is a complex function of variables describing this process. Generally, the heat transfer coefficient is a function of the fluid flow, component (probe) shape and dimensions,

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* Crude soybean oil is refined by a series of processes to remove impurities that affect the taste, smell, appearance, and stability of the oil. The refining processes involve degumming, alkali refining, bleaching, and deodorization. However, it is important to note that it is possible that the thermal treatment during the purification process may actually lead to peroxide formation and result in poorer oxidative stability when subjected to elevated temperatures during use (Ref 27).
** Historically, the earliest report of using a version of this test was by Tagaya and Tamura (Ref 25) who developed the test based on the Indiana Stirring Oxidation Test (Ref 31).
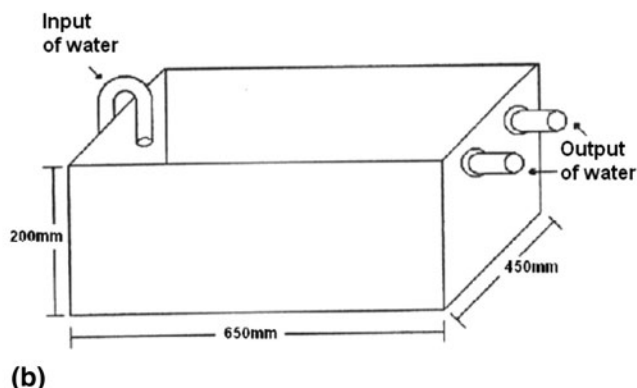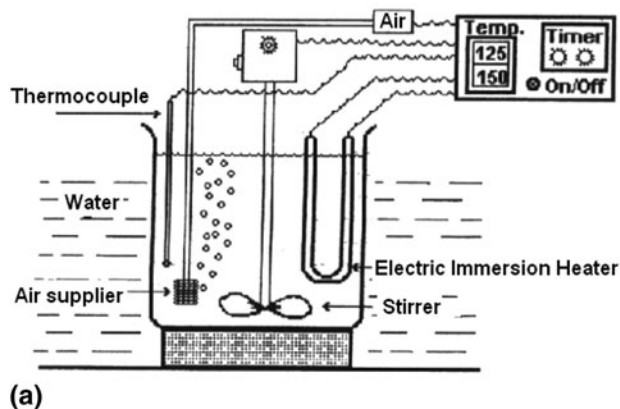
Fig. 1 Schematic illustration of the accelerated oxidation system: (a) heating, agitation, and blow air into the oil sample; (b) refrigeration system (Ref 30)

temperature, and physical properties of the liquid: thermal conductivity, specific heat capacity, density, and viscosity. The heat transfer coefficient can be defined as the quantity of heat transferred per unit time per unit area of a surface when the difference of temperatures between the surface and liquid equals one degree absolute. Since quenching processes are actually heat transfer processes, the heat transfer coefficient is an excellent single parameter for quenchant characterization.

In this work, the commercial code HT-MOD[†] (Heat Treating Modeling) was used to simulate the heat treatment process (Ref 32). This code is also used to calculate heat transfer coefficients as a function of time by solving an inverse heat transfer problem. The model is based on a numerical optimization algorithm which includes a finite element module for calculating, with respect to time and space, the temperature distribution and its coupled microstructural evolution. In this case, since an Inconel 600 probe was used that does not undergo microstructural phase transformation, the differential problem solved is only the heat conduction (Eq 1) in a cylindrical probe:

$$\nabla \cdot (k(\vec{r}, T) \cdot \nabla T) = c(\vec{r}, T)\rho(\vec{r}, T)\frac{\partial T}{\partial t} \qquad \text{(Eq 1)}$$

where $k(\vec{r}, T), c(\vec{r}, T)$, and $\rho(\vec{r}, T)$ denote the thermal conductivity, the specific heat, and the density of the material, respectively, as dependent of the position $(\vec{r})$ and temperature T. $T(\vec{r}, t)$ is subjected to the initial condition

$$T(\vec{r}, t = 0) = T_0(\vec{r}); \quad \vec{r} \in \Omega \qquad \text{(Eq 2)}$$

and the following boundary conditions at the surface of the steel probe:

$$-k\frac{\partial T}{\partial n} = h_i(T - T_{qu})\text{on}\Gamma_i, i = 1, \ldots, p \qquad \text{(Eq 3)}$$

where $h_i(T)$ is the heat transfer coefficient corresponding to different portions of the boundary Γ and T_{qu} is the quenchant temperature. Each one of these p cooling zones has a time-dependent heat transfer coefficient that varies strongly along

each partial boundary, depending on the heat transfer mechanism (vapor blanket, nucleate boiling, convective cooling) that governs the energy flow.

Provided that the temperature change inside the component and on its surface is measured, it is possible to solve the inverse heat transfer problem to determine the time variation of the heat transfer coefficients which best satisfies production demands. The time dependence of the heat transfer coefficient can be approximated by polygonal functions, each one defined by a set of parameters $h_i^{(r)}(r = 1, \ldots, p; \quad i = 1, \ldots, q)$.

On calling T_k^m, the measured temperature, and T_k^c, the numerically calculated temperature at those points, one can pose the problem of obtaining the values of the heat transfer coefficients h_i that minimize the function:

$$S = S\left(h_i^{(r)}\right) = \sum_{k=1}^{n} \left(T_k^m - T_k^c\right)^2 = \min \qquad \text{(Eq 4)}$$

n being the total number of measured temperatures, i.e., the number of points times the number of measurements at each point.

The selection of the initial values for these coefficients and of the quantity and length of the time intervals was sample-dependent. The mean square difference between the measured and calculated temperatures obtained after optimization of the heat transfer coefficients was about 1 °C. Table 2 summarizes the thermo-physical properties of INCONEL 600 used for this work (Ref 33).

4. Discussion

4.1 Thermal-Oxidation Test Results

In view of the relatively poor inherent oxidative stability of soybean oil and the commercial interest in using this commonly available base stock for industrial fluid formulation, it was selected for a subsequent preliminary screening test using two antioxidants, Irganox L 57 and Irganox L 109. The thermal-oxidation test described in the EXPERIMENTAL Section was used and the degree of oil oxidation was followed throughout the duration of the test by measuring the resulting fluid kinematic viscosity with respect to oxidation time as shown in

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[†] HT-MOD is a commercial code which is available from KB Engineering S.R.L.; Florida 274, Piso 3, Of. 35 (1005) Buenos Aires, *Argentina*; Tel:54-11 4326-7542; Fax: 54-11 4326-2424; Internet: http://www.kbeng.com.ar.

Table 2 Temperature-dependent thermo-physical values for INCONEL 600

Thermal conductivity		Specific heat		Density	
Temp., °C	K, W/m·K	Temp., °C	c, J/kg·K	Temp., °C	ρ, kg/m³
50	13.4	50	451	20	8400
100	14.2	100	467	100	8370
150	15.1	200	491	200	8340
200	16.0	300	509	300	8300
250	16.9	400	522	400	8270
300	17.8	500	533	500	8230
350	18.7	600	591	600	8190
400	19.7	700	597	700	8150
450	20.7	800	602	800	8100
500	21.7	900	611	900	8060
700	25.9				
900	30.1				
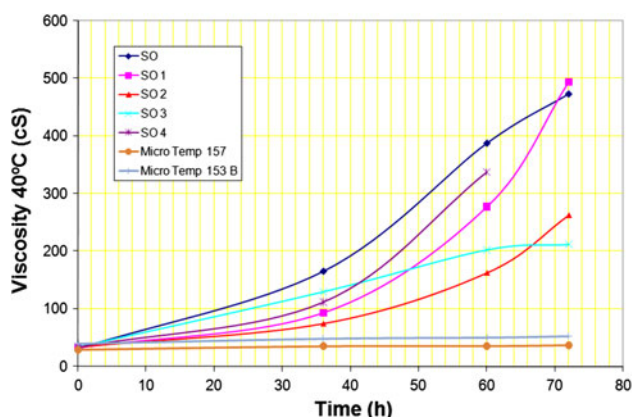
Fig. 2 Comparison of the ability of Irganox L 57 and Irganox L 109 and their blends to stabilize soybean oil using the Bashford and Mills oxidative stability test. Also shown for comparison are two commercial petroleum oil formulations: Microtemp 153B and Microtemp 157
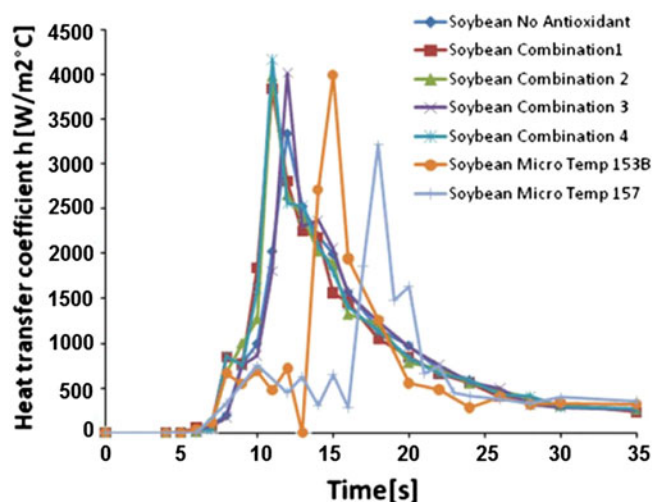
Fig. 2. For hydrocarbon-based fluids, thermal-oxidative degradation will result in an overall viscosity increase with respect to time, although in some cases, this increase may be preceded by a small viscosity decrease due to initial hydrocarbon chain cleavage before an exponential increase in viscosity.

Although there was some variation of the fluid viscosity upon addition of the antioxidants, the differences were relatively minimal. Throughout the aging test, the results obtained show that both Irganox L 109 and Irganox L 57 exhibited better stabilization than their blends. For most of the test, Irganox L 109 exhibited somewhat better performance than Irganox L 57. At the conclusion of the test, however, there was an inversion between 60 and 70 h where Irganox L57 seemed to stabilize with little additional increase in viscosity, whereas the Irganox L 109 continued on its exponential viscosity increase. This is an important observation and unfortunately the test was not continued beyond 70 h. To draw a definitive conclusion, longer test durations would be required.

One of the reasons for evaluating blends of Irganox L 57 and Irganox L 109 was to search for potential synergies. Based on these limited studies, no positive synergies by blending both antioxidants were observed.

Figure 2 also shows that the performance of neither Irganox L 57 nor Irganox L 109 exhibited the much better performance obtained with both commercial petroleum oil quenchants: Microtemp 157 or Microtemp 153B. Clearly, substantially greater stabilization of the soybean oil candidates is necessary if oxidative stability that is comparable to that exhibited by petroleum oils is to be achieved.

4.2 Effect of Aging on Heat Transfer Coefficients during Quenching

Any hyrdrocarbon-containing quenchant will exhibit some finite propensity to oxidize over time when subjected to high interfacial temperatures such as those encountered upon immersion of austenitized steel ($\approx$ 850 °C) and oxygen (air). The degree and rate of oxidation will vary with respect to the chemical structure of the hydrocarbon moiety; the presence, structure, and concentration of antioxidants; the presence of contaminants such as water and metals including copper and iron; and total load size and frequency of use of the quench bath
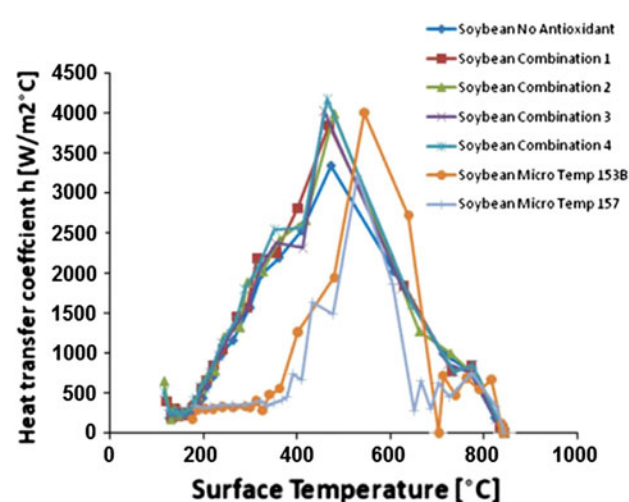
Fig. 3 Heat transfer coefficient as a function of time (left) and as a function of surface temperature (right), calculated for the seven oils; time of degradation 0 h and bath temperature 40 °C

Fig. 4 Heat transfer coefficient as a function of time (left) and as a function of surface temperature (right), calculated for the seven oils; time of degradation 24 h and bath temperature 40 °C
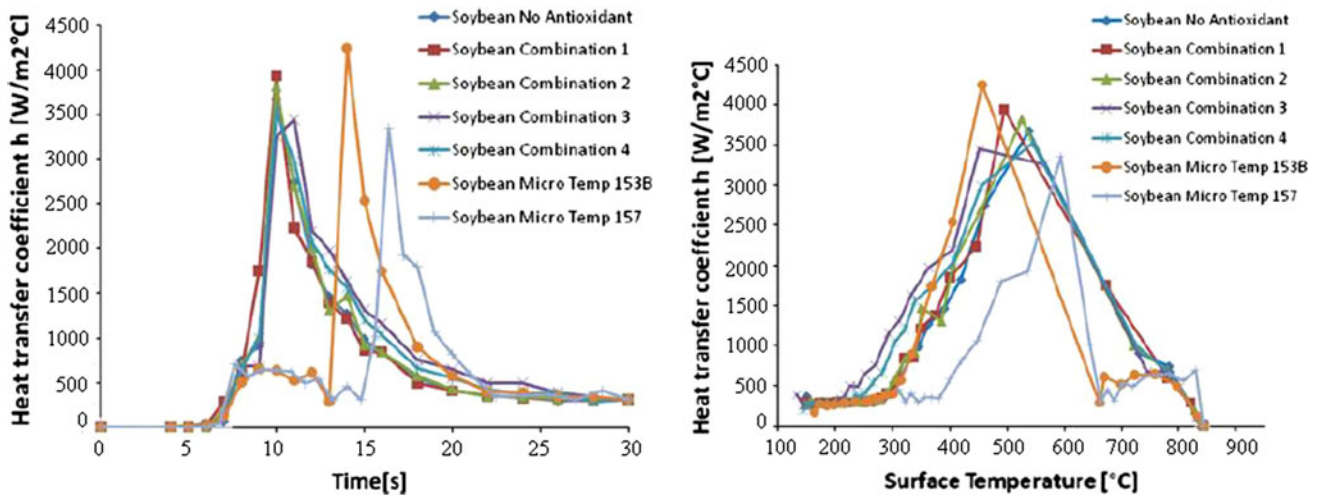

Fig. 5 Heat transfer coefficient as a function of time (left) and as a function of surface temperature (right), calculated for the seven oils; time of degradation 48 h and bath temperature 40 °C
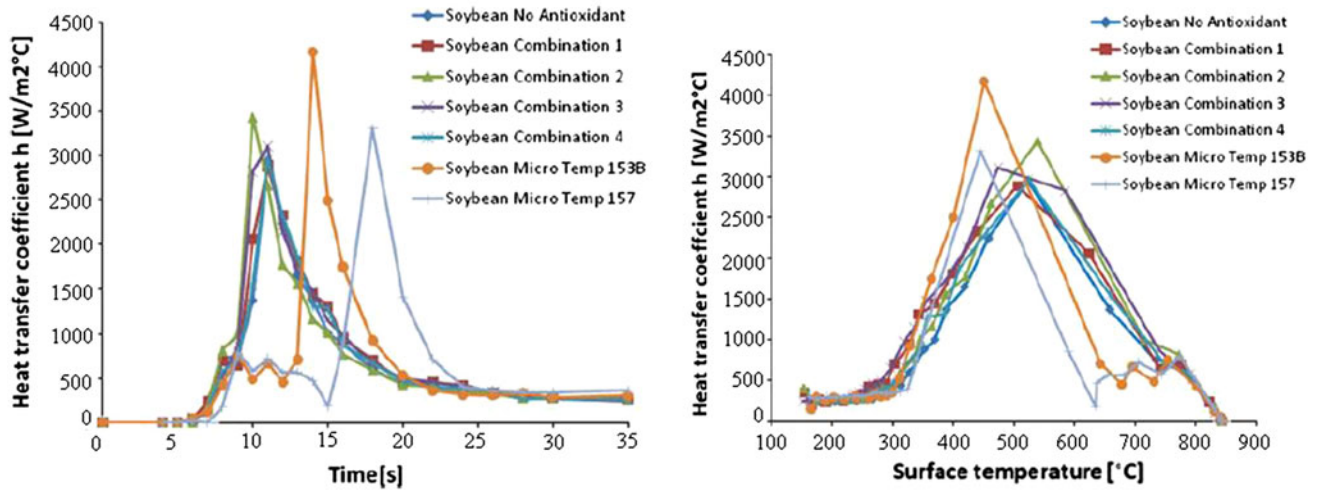
among other variables (Ref 35-37). Therefore, some net increase in quenchant viscosity over time is expected and since viscosity is an important variable in heat transfer, some variation in heat transfer rates is also expected (Ref 34). The question is how great is the variation and what would be the relative variation when different quench media are compared.

For this work, cooling curve analysis was performed according to the ASTM D6200 test procedure at a quenchant bath temperature of 40 °C with the different soybean oil formulations and petroleum oil-based quenchants shown in Table 1. The heat transfer coefficients as a function of time and surface temperatures data are shown in Fig. 3-6.

As expected, the heat transfer coefficient versus time data showed that the conventional, unaccelerated MicroTemp157 petroleum oil-based quenchant exhibited significantly longer film boiling behavior compared to the fast, accelerated MicroTemp 153B quench oil. This behavior is due to the addition of additives that rupture the vapor blanket that forms around the hot-metal surface upon immersion of the metal into

the quenchant. Significantly, this work confirm an earlier reported cooling curve behavior showing that vegetable oils do not exhibit this behavior (Ref 38).

Table 3 summarizes the maximum heat transfer coefficient, surface temperature, and cooling time where this occurs. The maximum heat transfer coefficient for fresh, unoxidized soybean oil is significant less than soybean oil containing antioxidants and is comparable to the unaccelerated MicroTemp 157 quench oil which interestingly is less than the accelerated petroleum oil, MicroTemp 153B. Also, while the addition of an antioxidant or antioxidant mixture does significantly increase the maximum heat transfer coefficient relative to fresh, unstabilized soybean oil, the amount of increase with respect to different antioxidant additions is not very large.

The effect of antioxidant addition on surface temperature and time where the maximum heat transfer coefficient occurs does not vary greatly for soybean oil. However, the surface temperature where the maximum heat transfer coefficient occurs is similar for both MicroTemp 153B and MicroTemp 157, but the time where
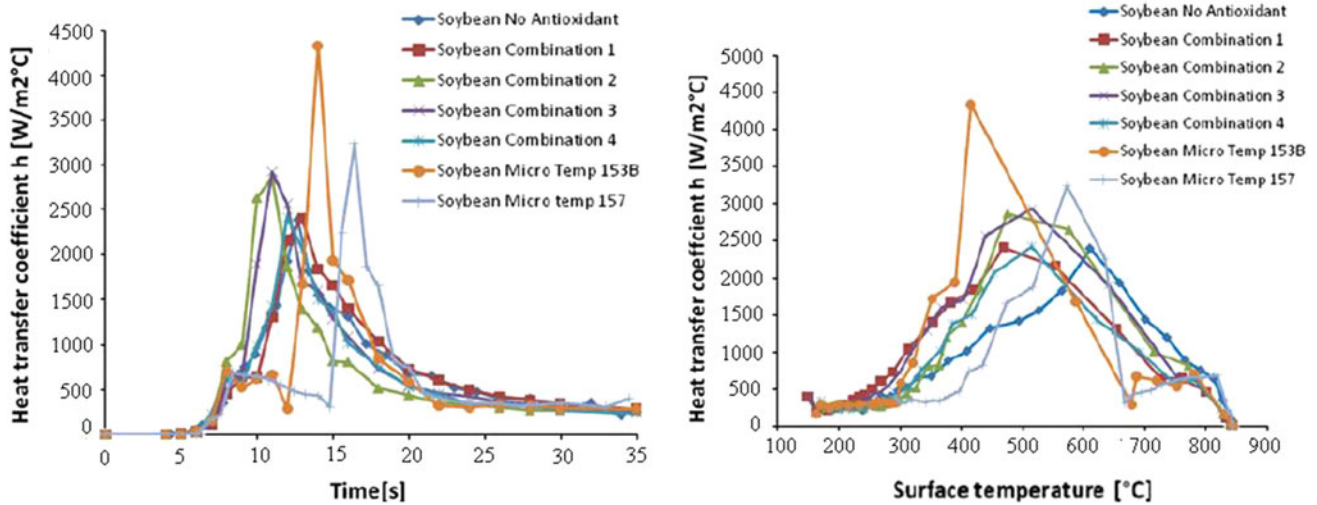
Fig. 6 Heat transfer coefficient as a function of time (left) and as a function of surface temperature (right), calculated for the seven oils; time of degradation 60 h and bath temperature 40 °C

Table 3 Summary of quenching heat transfer parameters obtained from cooling curve analyses on the different soybean oil formulations and commercial petroleum-based quenchants

| | Aging time(b) | | | | | | | | | | | |
| | 0 h | | | 24 h | | | 48 h | | | 60 h | | |
Quenchant(a)	h_{max}	T_S	t_{hmax}	h_{max}	T_S	t_{hmax}	h_{max}	T_S	t_{hmax}	h_{max}	T_S	t_{hmax}
SO	3340	473	12	3672	538	10	2946	522	11	2391	610	13
SO1	3840	469	11	3940	494	10	2886	505	11	2402	469	13
SO2	3989	480	11	3817	526	10	3440	539	10	2859	475	11
SO3	4020	458	12	3450	451	11	3113	474	11	2920	515	11
SO4	4170	465	11	3518	544	10	2966	524	11	2416	515	12
MicroTemp 153B	4000	544	15	4250	456	14	4170	450	14	4330	415	14
MicroTemp 157	3210	529	18	3355	593	16.5	3310	444	18	3243	572	16.5

(a) The quenchant designations indicate: SO, uninhibited soybean oil; SO1, 0.3% Irganox L 57 + 0.7% Irganox L 109; SO2, 1% Irganox L109; SO3, 1% Irganox L57; SO4, 0.5% Irganox L 57 + 0.5% Irganox L 109; MicroTemp 153B is a commercial accelerated petroleum oil quenchant and MicroTemp 157 is a commercial unaccelerated petroleum quenching oil. (b) The parameters shown here are maximum heat transfer coefficient for the overall cooling process—h_{max} [W/m²·°C]; Surface temperature at h_{max} — T_S (°C); and the time at h_{max} — t_{hmax}

this event occurs is somewhat greater for both oils than the soybean oil formulations with the unaccelerated MicroTemp 157 exhibiting the longest time—as expected.

Aging of the different oils showed a general decrease in the maximum heat transfer coefficient with respect to time as would be expected with the overall fluid viscosity increases illustrated in Fig. 2. Generally, the amount of decrease was the greatest for the least stable oils. There was no clear correlation in the source temperature and cooling time where the maximum heat transfer coefficient occurred, which may be due to the overall reported variability of the ASTM D6200 method.

Compared to the variation of the maximum heat transfer coefficients observed for the soybean oil fluids with increasing aging time, neither petroleum oil-based quenchant—Micro-Temp 153B or MicroTemp 157—exhibited any significant difference in cooling behavior throughout the test, which is

reasonable in view of the minimal viscosity change observed throughout the aging test for both oils as shown in Fig. 2.

5. Conclusions

The effect of the addition of two commercial antioxidants, Irganox L57 and Irganox L109, on the thermal-oxidative stability of soybean oil was studied. The results showed that both L57 and L109 did inhibit the thermal-oxidative degradation process compared to uninhibited soybean oil. However, even though the concentration of these antioxidants was not optimized, neither produced a stabilized soybean oil that even roughly approximated the stability of a commercial, fully formulated petroleum-based quenchant.

Cooling curve analysis showed that soybean oil, with or without the addition of an antioxidant, exhibited any significant full-film boiling behavior, whereas the petroleum oil quenchants did exhibit such behavior and, as expected, the accelerated petroleum oil (MicroTemp 153B) exhibited the less stable vapor blanker cooling behavior compared to the conventional MicroTemp 157 quench oil.

The cooling curves study also showed that the maximum heat transfer coefficient was greater (faster) for soybean oil containing antioxidants compared to uninhibited soybean oil. In addition, the maximum heat transfer coefficient decreased faster (due to fluid viscosity increase attributable to the thermal-oxidative degradation process) than the inhibited soybean oil fluids, with Irganox L109 and Irganox L57 being the most effective. However, this performance fell considerably short of that observed for both fully formulated petroleum oil quenchants, the maximum heat transfer coefficients of which did not change significantly throughout the test.

As a result of this work, it is concluded that although the addition of antioxidants does stabilize soybean oil, substantially better performance is required if vegetable oils are to be effective functional equivalents to petroleum oil formulations. This may be done by selecting different vegetable oil compositions with less unsaturation, by genetic modification of soybean seed oils or by chemically modifying and stabilizing the vegetable oil structure.

Acknowledgments

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