

Low-Temperature Filterability Properties of Alternative Diesel Fuels from Vegetable Oils

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ABSTRACT

Methyl esters from vegetable oils have many characteristics that make them attractive as a fuel for combustion in compression ignition (diesel) engines. Recent research has shown that the issue of cold flow properties should be resolved before methyl esters will be attractive as an alternate fuel in moderate temperature climates. In North America, fuel systems powered by petroleum middle distillates develop operability problems when overnight ambient temperatures drop below -15°C (5°F). In contrast, methyl esters from soybean oil develop similar problems at temperatures near 0°C (32°F). This work examines expected operability limits for methyl esters through evaluation of their low-temperature filterabilities. Low-temperature flow test (LTFT) and cold filter plugging point (CFPP) results showed nearly linear dependence with respect to cloud point (CP). Under most conditions, CFPP was nearly equivalent to LTFT as a predictor of operability limits for methyl esters, with respect to conditions in North America. Slight improvement in filterability with respect to CP was observed when methyl esters were treated with "off-the-shelf" diesel fuel cold flow additives. Results from differential scanning calorimetry (DSC) melting curves also showed a correlation with CP. Finally, admixtures of up to 30 vol% methyl tallowate in methyl soyate showed no significant deviation with respect to low-temperature filterability or CP. Overall, this work demonstrates that emphasis on reducing CP remains the key to developing methyl ester diesel fuels with improved low-temperature flow properties.

Key Words: DSC, Methyl esters, Physical properties, Soybean oil, Tallow.

INTRODUCTION

Methyl esters from transesterified vegetable oils or animal fats are attractive as a fuel for combustion in direct injection compression ignition (diesel) engines (Schwab *et. al.*, 1987; Sims, 1985). With respect to petroleum middle distillates, methyl esters offer improved emission quality such as reduced smoke opacity, particulates, unburned hydrocarbons and carbon monoxide (Masjuki *et. al.*, 1993; Scholl and Sorenson, 1993). Methyl esters also offer safer handling and storage options owing to their relatively high flash points and environmentally innocuous nature. Thus, methyl esters represent an attractive alternative fuel in market areas such as underground mining, marine applications and vehicle fleets operating under Energy Policy Act (EPACT) or Clean Air Act guidelines.

Before methyl esters become viable as a fuel in the North American transport industry, the crucial issue of improving their low-temperature flow properties must be resolved. Distillate fuels typically develop operability problems such as wax settling and plugging of filters and fuel lines when overnight temperatures approach -10 to -15°C (Chandler *et. al.*, 1992; Lewtas *et. al.*, 1991; Owen and Coley, 1990). Likewise, methyl esters from soybean oil (SME) develop similar problems at temperatures approaching 0°C , a significantly higher temperature. Dunn and Bagby (1995) showed that blending SME with No. 2 diesel fuel yields a nearly linear reduction in cloud

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point (CP); however, that study also pointed out that blend ratios should not exceed 20 vol% SME to prevent CP from increasing to a point exceeding -10°C .

Studies conducted by Dunn et. al. (1995; 1996) have shown a nearly linear relationship between CP and low-temperature filterability of distillate/methyl ester blends. That is, cold-temperature operability limits predicted by standard filterability tests were directly proportional to corresponding CP. This trend was observed for formulations containing 10 to 100 vol% methyl esters. These studies pointed out that approaches to improving the low-temperature flow properties of blends should concentrate on reducing CP. This was an important result because traditional methods for improving cold flow of distillates have focused on depressing pour point rather than CP (Brown and Gaskill, 1990; Gwen and Coley, 1990). Thus, at blend ratios as small as 10 vol% methyl esters, the dynamics of wax agglomeration as temperatures drop below CP are greatly altered with respect to improving cold flow properties.

This work is an investigation of low-temperature flow properties of neat methyl esters. Emphasis was placed upon non-blended methyl esters for two reasons. The apparent effects of relatively small volumes of methyl esters in middle distillates support the argument that low-temperature flow properties of blends may be dominated by the presence of methyl esters. In other words, techniques that improve the cold flow properties of methyl esters are likely to be effective in blends. The second, more practical reason arises from the notion that some market areas such as marine and underground mining may not tolerate fuels formulated from distillates.

Filterability results from both low-temperature flow test (LTFT) and cold filter plug point (CFPP) studies were compared statistically with corresponding CP data. Comparisons were made between SME, methyl ester admixtures with up to 30 vol% tallowate methyl esters (TME) in SME and SME treated with cold flow improver additives. Finally, low-temperature properties were compared with results from heating curves from differential scanning calorimetry (DSC).

METHODS AND MATERIALS

Soyate methyl esters (SME) were from Interchem Environmental (Overland Park, KS) via the National Biodiesel Board (Jefferson City, MO). Samples were double-distilled at the refinery to remove nearly all traces of glycerine. Gas chromatography analysis showed 10.6 wt% hexadecanoate (C16:0), 4.3 % octadecanoate (C18:0), 24.4 % octadecenoate (C18:1), 51.9 % octadecadienoate (C18:2) and 8.2 % octadecatrienoate (C18:3). Tallowate methyl esters (TME), Kemester 143, were from Witco (Memphis, TN). Gas chromatography of TME showed 0.2 wt% tetradecanoate(C14) 28.7 %C16:0, 2.6%hexadecanoate(C16:1), 20.8%C18:0,43.0% C 18: 1, 4.3 % C 18:2 and 0.4 % C 18:3. Other physical properties of SME and TME were reported earlier (Dunn and Bagby, 1995)

Six cold flow improvers were tested in this work. 8500 Winterflow was from Starreon Corp. (Englewood, CO); DFI- 100 and DFI-200 were from Du Pont (Wilmington, DE); Hitec 672 was from Ethyl Petroleum Additives (Rolling Meadows, IL); OS 10050 was from SVO Enterprises (Eastlake, OH); and Paramins was from Exxon (Abington, Oxfordshire, England). The additives generally consisted of proprietary mixtures of ethylene/vinyl acetate copolymers and naphthenic distillates.

Apparatus and methodologies for measuring CP, CFPP and LTFT have been described previously (Dunn and Bagby, 1995). Differential scanning calorimetry (DSC) was carried out with a TA Instruments (New Castle, DE) DSC 2910 and a TA 2100 PC-based controller. The measurement cell was purged with low-pressure nitrogen gas and fitted with a heat exchanger with liquid nitrogen coolant for sub-ambient scans. For each scan, approximately 10 mg of sample was hermetically sealed in an aluminum pan and tested against an empty pan. Samples were cooled and held isothermally at -100°C for 10 min before ramping (heating) to 40°C at $5^{\circ}\text{C}/\text{min}$. Heating curves were analyzed for onset temperatures located at the completion of the higher melting (p-form) peaks.

RESULTS AND DISCUSSION

Filterability Results

Table 1 is a summary of CFPP and CP data collected in this work. For purposes of brevity, only 10 formulations appear in this table. In total, 44 formulations consisting of SME, SME/TME admixtures (5, 10, 20 and 30 vol% TME) and additive treated (500, 1000, 1500 and 2000 ppm) SME and admixtures were examined. Least-squares regression yielded the following expression:

$$\text{CFPP} = -1.9 + 0.3964 * (\text{CP}). \quad (1)$$

The standard error of the y-estimate (a) was 1.9, corresponding to a variance from residuals (σ^2) of 3.6555, and the regression coefficient (R^2) was 0.1629. The relatively small R^2 value indicated that a significant degree of deviation exists between Eq. 1 and the data.

TABLE 1 | Cloud Point (CP) and Cold Filter Plugging Point (CFPP) Results. Additive Loading was 2000 ppm.

Admixture	Additive	CP (°C)	CFPP (°C)
SME†	None	0	-3
SME	8500 Winterflow	-1	-2
SME	DFI- 100	-1	1
SME	DFI-200	-1	-4
SME	Hitec 672	-2	-2
SME	OS 110050	-3	-1
SME	Paramins	-1	1
4:1 (vol) SME/TME‡	None	2	0
4:1 (vol) SME/TME	DFI- 100	4	0
4:1 (vol) SME/TME	Hitec 672	2	-5

† SME = methyl esters from soybean oil

‡ TME = methyl esters from tallow

Dunn *et al.* (1995; 1996) showed that CFPP of distillate/methyl ester blends may be linearly correlated with CP by the following:

$$\text{CFPP} = -2.2 + 1.0276 * (\text{CP}), \quad (2)$$

when $-30 < \text{CP} < 10^\circ\text{C}$. Comparing Eq. 2 with non-blended methyl esters data in this work yields $\sigma = 2.4$ and $R^2 = 0.3150$. Although σ was comparable to that for Eq. 1, R^2 shows a significant degree of scatter in data for non-blended methyl esters with respect to distillate/methyl ester blends. Even so, results show that an equation derived earlier for blends (Eq. 2) may be sufficient for estimating CFPP behavior for non-blended methyl esters.

Figure 1 is a plot of LTFT vs. CP data collected from 32 formulations (note, several points overlap each other). Least-squares regression yielded the following expression:

$$\text{LTFT} = -2.0 + 0.2994 * (\text{CP}). \quad (3)$$

where the point representing TME (CP = 17°C ; LTFT = 20°C) was omitted from regression analysis. For Eq. 3, $a = 1.7$ and $R^2 = 0.0849$; thus, this equation is not a very efficient means for

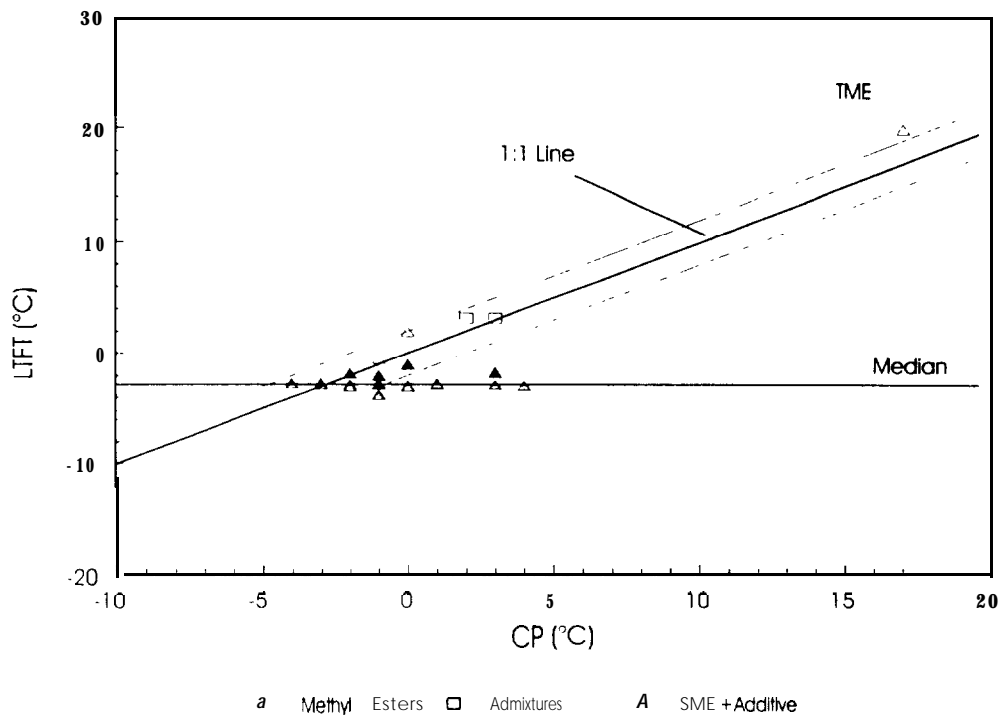


Figure 1. Low-temperature flow test (LTFT) results plotted with respect to cloud point (CP). Additive loading was 2000 ppm. Median LTFT = -2.8°C; dashed lines are American Society for Testing Materials (ASTM) limits for repeatability (ref., ASTM, 1991. Method D4539). Methyl esters = SME or TME; Admixtures = SME/TME mixtures. See Table 1 for other abbreviations.

predicting LTFT of non-blended methyl esters. Aforementioned studies on distillate/methyl ester blends yielded the following:

$$LTFT = -2.4 + 0.8140 \cdot (CP) \quad (4)$$

where comparison with data from Fig. 1 yielded $a = 2.0$ and $R^2 = -0.3576$. Although σ was comparable, Eq. 4 significantly reduces the degree of scatter with respect to R^2 .

Overall, expressions derived earlier for distillate/methyl ester blends may be sufficient for estimating LTFT or CFPP from CP data for non-blended methyl esters. However, a relatively large degree of scatter between filterability and CP will impose limitations on predicting LTFT or CFPP from linear correlations. The data in Fig. 1 have been broken down by composition with respect to methyl esters (SME and TME), methyl ester admixtures and additive treated SME. This breakdown revealed two distinct trends. Comparison with the 1:1 line (LTFT = CP) showed that a minimum of 20 points lie within guidelines for repeatability ($\pm 2^\circ\text{C}$) specified in the American Society for Testing Materials (ASTM) standard method for measuring LTFT (ASTM, 1991. D4539). This trend agrees well with those noted for distillate/methyl ester blends. On the other hand, examination of SME + Additive data shows that 27 data points lie within $\pm 2^\circ\text{C}$ of the median LTFT (-2.8°C , based on the entire data set). Thus, treatment of methyl esters with additives showed a large probability of greatly reducing the dependence of LTFT on CP. Studies by Dunn *et al.* (1996) showed that the correlation between LTFT and CP becomes increasingly unstable as CP increases for additive-treated distillate/methyl ester blends. Although results from this work agree with that general trend, the effects of additives on reducing the dependence of LTFT on CP appear to be confined to non-blended methyl esters.

Statistical analysis of filterability-CP data was carried out by paired two-sample t-testing. Testing against the mean hypothetical difference ($CP - CFPP = 0$) showed very little probability for correlation (<0.00001). Earlier work by Dunn *et al.* (1995; 1996) showed a relatively large probability that the difference for distillate/methyl ester blends was ($CP - CFPP = 2$). Testing non-blended methyl esters against this difference a significant (0.811) probability that this hypothesis was acceptable. Thus, it is **likely** that CFPP and CP of both blended **and** non-blended methyl esters may be correlated by an “off-setting” constant equal to 2°C.

Testing against the difference ($CP - LTFT = 0$) also showed very little probability for correlation (<0.0001). Again referring to work by Dunn *et al.* (1995; 1996), probabilities for this correlation were 0.579 for additive treated blends and 0.943 for non-treated blends. Analogous to CFPP results in the current study, t-testing the difference ($CP - LTFT = 2$) gave a relatively high probability (0.641) for accepting that hypothesis. **Thus**, with respect to non-blended methyl esters, this work shows that an off-setting difference between **LTFT** and CP may exist and that this difference may be identical to that noted for CFPP and CP. In other words, it is likely that filterability data for non-blended methyl esters does not greatly depend on the particular methodology employed, regardless of whether additives were present. Paired two-sample f-tests for the difference ($CFPP - LTFT = 0$) yielded a probability of 0.382 for accepting this hypothesis. In practical terms, this work shows that CFPP data may be substituted, under most conditions, for **LTFT** data when evaluating low-temperature operability limits of fuel systems powered by **non-blended** methyl esters in North America. This may be a significant conclusion owing to advantages favoring CFPP with respect to timeliness, user friendliness and availability of experimental apparatus (Dunn and Bagby, 1995).

DSC Heating Curves

Completion of melt type crystallization onset temperatures (T_c) were determined from analysis of high melting (p-form) peaks. Figure 2 is a graphical demonstration of how T_c was determined with respect to a hypothetical DSC heating curve based on methyl esters studied in this work. Essentially, T_c was inferred **from** the point of intersection of lines drawn tangent to the peak and baseline. Lee *et al.* (1995) studied **alkyl** esters from soybean oil **and** determined onset temperatures **from** the point of convergence to the baseline (T_c). They also reported that **analysis** of heating curves gave very reproducible results with respect to ramp rate. Cebula and Smith (1991) observed similar results for triglycerides. These studies noted that onset temperatures from cooling curves depended greatly on ramp rate (1 to **20°C/min**), attributing the variances to the dependence of nucleation on cooling rate. Consequently, DSC heating curves were selected for analysis in this work.

Figure 3 is a plot of T_c vs. CP data collected from formulations representing a **cross-section** of those evaluated in filterability studies discussed above. In general, this cross section featured SME, TME, admixed methyl esters and **SME + 2000 ppm additives** (8500 **Winterflow** and **DFI-200**). Each T_c value plotted in Fig. 3 represents a **mean** taken **from** 3 thermal analyses, one each with a fresh sample. Confidence intervals in Fig. 3 were with respect to a **significance** level of 0.10 ($\alpha = \pm 0.05$). In general, intervals were in the range ± 0.02 to $\pm 0.60^\circ\text{C}$, two exceptions being the SME + additive points whose intervals were ± 0.70 to $\pm 1.25^\circ\text{C}$. Thus, statistical analysis of **the** experimental method for **determining** T_c , showed very good to excellent reproducibility.

Similarly, each CP value in Fig. 3 represents a mean taken **from** three replicate measurements, in accordance with the ASTM standard method for **measuring** CP (ASTM, 1991, D2500). Variances (σ^2) were in the range 0 to 1, producing a pooled variance of 0.2917 for a standard deviation of only 0.5°C . Confidence intervals with respect to $\alpha = \pm 0.05$ were in the range ± 0.0 to $\pm 1.7^\circ\text{C}$, that is, intervals ranged **from well** to just within guidelines specified for reproducibility ($\pm 2^\circ\text{C}$) in the ASTM methodology. Thus, the experimental method for determining CP yields **sufficiently** reproducible results with respect to ASTM guidelines.

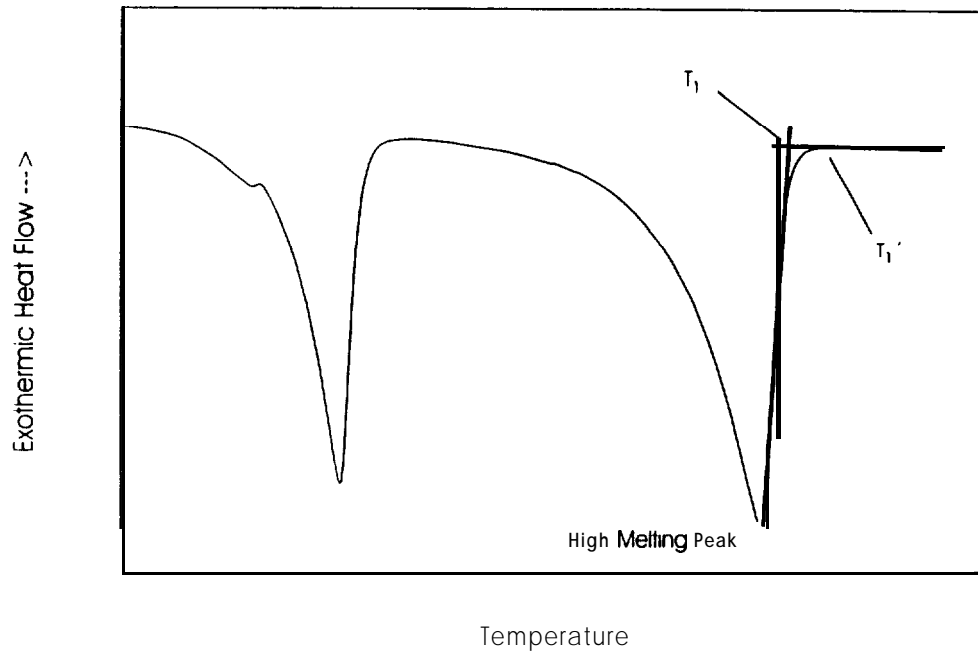


Figure 2. Measurement of crystalliion onset temperature (T_1) **from** a differential scanning calorimetry (DSC) heating curve. Heating scan is hypothetical; T_1' = point of deviation **from** baseline.

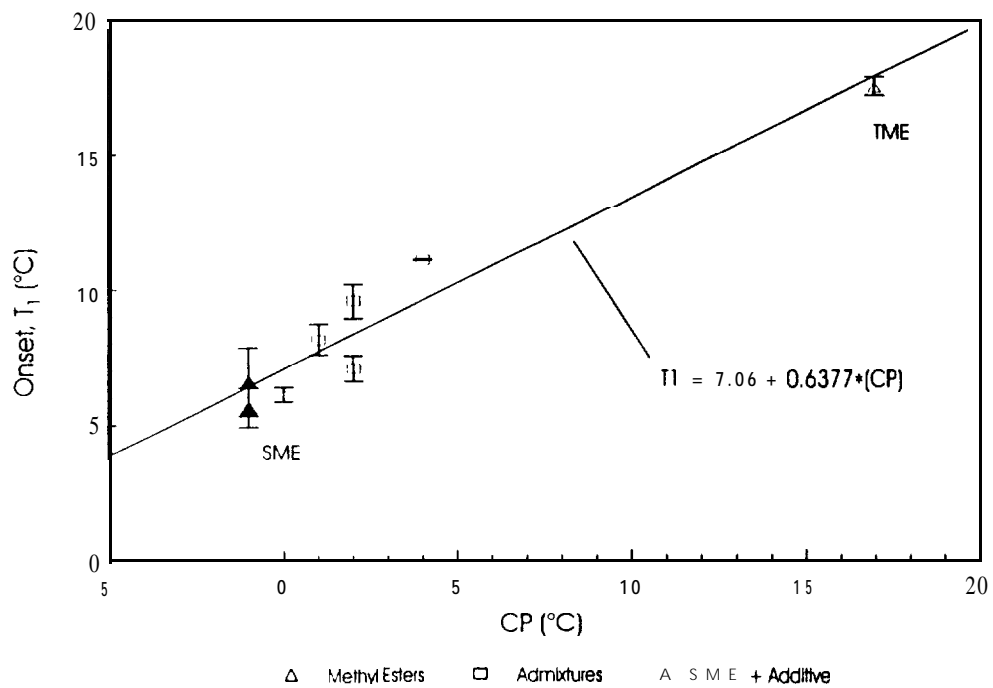


Figure 3. T_1 results plotted with respect to CP. Additive loading was 2000 ppm. Bars are confidence intervals with **significance** level = ± 0.05 . Methyl esters = **SME** or **TME** and **Admixtures** = **SME/TME** mixtures. See Table 1 and Figure 2 for other abbreviations.

In general, studies of liquid petroleum products have demonstrated the **usefulness** of DSC for measuring CP. Heino (1987) reported that a very good correlation exists between CP and crystallization onset temperature determined **from** cooling curves for middle distillates. That work showed that CP could be inferred **from** either T, or T₁' (see Fig. 2) with greater precision ($\pm 0.7^{\circ}\text{C}$) than that called for in ASTM standard methodology. Others (**Redelius**, 1987; Claudy et al., 1986) have reported similar results.

Least-squares regression of T, vs. CP data for non-blended methyl esters yielded the following expression:

$$T, = 7.06 + 0.6377*(CP) \quad (5)$$

which is represented by the solid line in Fig. 3. Although the line intersects only three of the confidence intervals for T,, it was within only 0.10-1.47°C of doing so for the remainder. Variance with respect to residuals (σ^2) was 1.1973; thus, the standard error of the y-estimate (σ) was only 1.09. Also, R² was 0.9212, a value **sufficient** to accept the hypothesis that a linear correlation exists between CP and T, of non-blended methyl esters.

This correlation is in good agreement with results **from** reference studies of petroleum products. **Thus**, a practical result **from** this work may be the development of a method for predicting CP of methyl esters directly **from** DSC heating curves. Inverting the data in Fig. 3 (to CP vs. T,) and re-analyzing by least-squares regression yielded the following:

$$Cp = -10.1 + 1.4623*(T,) \quad (6)$$

where $\sigma^2 = 2.7456$ (R² was identical to that for Eq. 5). This expression allows prediction of CP data **from** DSC heating curves with an estimated error of $\pm 1.6^{\circ}\text{C}$. This level of precision is superior to that called for in ASTM standard methodology. Thus, **if** T₁ can be measured accurately **from** DSC heating curves, then Eq. 6 may be employed to determine a CP corresponding to T,, with the results feasibly more accurate than those taken **from** the ASTM standard method.

Determination of CP **from** thermal analysis has many advantages over ASTM method D2500. The ASTM method is influenced by subjectivity because it calls for visual detection of haziness in the sample. Inferring CP **from** DSC curves greatly minimizes experimental error associated with visual detection. Thermal analysis offers additional benefits by providing important information on nucleation and crystallization. For example, at a ramp rate of 5°C/min, T, of SME was 6.16°C. At this temperature, crystallization in the form of nuclei with very small particle sizes is initiated. Unless a **sufficient** number of crystals with particle sizes in excess of 0.5 μm, they will not be visible in solution (Chandler et al., 1992; Lewtas et al., 1991). Thus, CP for SME does not occur until temperatures decrease below T, to a point near 0°C. DSC curves can be useful in characterization of polymorphism generally associated with solid-phase fats, oils and fatty acid derivatives (Hagemann, 1988; Sato, 1988), determination of thermodynamic properties such as enthalpy of fusion and examination of thermal history. **Finally**, DSC analyses may be **useful** in studying the chemical and physical nature of saturated and unsaturated methyl ester fatty acid tailgroups in relation to the kinetics of nucleation and crystallization. These studies should produce some insight into developing approaches to improve the low-temperature flow properties of alternative diesel fuels formulated with methyl esters **from** fats or vegetable oils.

CONCLUSIONS

Filterabilities of SME, TME and SME/TME admixtures without additives showed a linear correlation with CP. Statistical analysis showed that LTFT and CFPP may be estimated directly from CP by an offset constant of -2°C. In general, treatment with cold flow additives marketed

for petroleum middle distillates rendered LTFT nearly independent of CP. On the other hand, the correlation between CFPP and CP was not affected by the presence of cold flow additives, a result that was in agreement with those **from** likewise studies of **distillate/methyl** ester blends. Regardless of the presence of additives, results showed that **LTFT** was statistically equivalent to CFPP.

Crystallization onset temperature (T_c) may be measured with very good reproducibility by analysis of higher melting (p-form) peaks **from** DSC heating curves. For methyl esters and admixtures, CP correlated well with T_c. Thus, CP may be more accurately predicted by thermal analysis than with standard methodologies. This work shows that thermal analysis is crucial to the development of alternative diesel **fuels** from fats **and/or** vegetable oils in at least the following two areas: a) fundamental studies of the nucleation, growth and agglomeration as well as morphology of fatty methyl esters and other derivatives; and b) identification and **evaluation** of approaches for improving low-temperature flow properties of fatty methyl esters and other derivatives.

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