

# Physical Properties of Oleochemical Carbonates

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**ABSTRACT:** Carbonates are a class of compounds that have recently found increasing interest in commercial applications owing to their physical properties and relatively straightforward synthesis. In this work, physical and fuel properties of five straight-chain C<sub>17–39</sub> and three branched C<sub>17–33</sub> oleochemical carbonates were investigated. These properties included cetane number (CN), low-temperature properties, (kinematic) viscosity, lubricity, and surface tension. The carbonates studied had CN ranging from 47 to 107 depending on carbon chain length and branching. For the same number of carbons, the CN of carbonates were lower than those of FA alkyl esters owing to interruption of the CH<sub>2</sub> chain by the carbonate moiety. Kinematic viscosities at 40°C ranged from 4.9 to 22.6 mm<sup>2</sup>/s whereas m.p. ranged from +3 to below –50°C depending on the carbonate structure. High-frequency reciprocating rig testing showed the neat carbonates to have acceptable lubricity that improved as chain length increased. Finally, the carbonate's ability to influence cold-flow properties in biodiesel (methyl soyate) and lubricity in low-lubricity ultra-low sulfur diesel were examined. The carbonates studied did not significantly affect cold flow or lubricity properties at concentrations up to 10,000 ppm (1 wt%). The properties of the carbonates resemble those of fatty alkyl esters with similar trends resulting from compound structure.

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**KEY WORDS:** Branched carbonates, cetane number, cloud point, dialkyl carbonates, Guerbet carbonates, linear carbonates, oleochemical carbonates, lubricity, pour point, viscosity.

Carbonates (esters of carbonic acid, H<sub>2</sub>CO<sub>3</sub>) represent a versatile class of organic compounds known to chemists for their interesting chemistry (1,2). Because of improved large-scale production processes, low M.W. short-chain aliphatic carbonates such as dimethyl and diethyl carbonate have seen renewed interest (3). Currently, they are being considered as oxygen-containing fuel additives to reduce vehicle exhaust emissions in gasoline engines (4,5), and they are also gaining popularity as nontoxic replacements for hazardous chemical reagents such as phosgene and dimethyl sulfate (6). Mid-chain dialkyl carbonates, containing alkyl chains between three and eight carbon atoms, represent a newer class of interesting lubricants and functional fluids with improved seal compatibility and toxicology (7,8) and are expected to gain importance and application.

Renewed interest in the use of fats and oils from vegetable and animal origin to prepare materials through sustainable chemistry has led to a revival in the development of biobased materials. Oleochemical carbonates, carbonic acid esters prepared from animal- and plant-based feedstocks, are among this movement, and their interesting properties find application as components in lubricants (7–9), cosmetics (10,11), and plasticizers (12). With regard to lubricant applications, the highly polar nature of the carbonate moiety enables it to adhere strongly to metal surfaces. Tribological testing suggests that carbonates have lubricity properties comparable to or better than mineral oil and traditional synthetic esters with similar viscosities (7). Unlike esters, the carbonate moiety decomposes to carbon dioxide and alcohols without formation of corrosive carboxylic acids typically observed in conventional synthetic ester lubricants (7,8).

It is well-known that structural features of fatty compounds such as chain length, degree of unsaturation, and branching affect performance properties of biodiesel such as nitrogen oxide (NO<sub>x</sub>) emissions, cetane number (CN), viscosity, lubricity, and low-temperature properties such as cloud point (CP) and pour point (PP) (13,14). Other functional groups, such as the hydroxyl group (OH), can also influence performance. For example, Geller and Goodrum (15) studied lubricity effects derived from specific FAME including the hydroxyl-containing methyl ricinoleate (15). At additive levels of 1% in diesel fuel they found that as unsaturation in the FA esters increased, lubricity properties improved. Additionally, they showed that the methyl ester of ricinoleate (C18:1 OH) increased lubricity in diesel fuel more effectively than its nonhydroxylated counterpart, oleic acid (C18:1).

Because some oleochemical carbonates possess low m.p., good miscibility with hydrocarbon oils, and high thermal-oxidative stability (8) it is of interest to evaluate their potential as fuel extenders or additives. Although a sizeable body of patent literature and commercial applications of dialkyl carbonates exists (1,2), to our knowledge the physical and fuel properties of oleochemical carbonates with regard to various industrial applications have not been systematically reported.

In the present work, the physical and fuel properties of a series of linear and branched oleochemical carbonates synthesized previously (16) were investigated. These properties include CN, m.p., kinematic viscosity, lubricity, surface tension, refractive index, and density. The carbonate properties determined in this study were found to be comparable to the properties of fatty alkyl esters because of the similarities between their structures.

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## EXPERIMENTAL PROCEDURES

**Materials.** FAME of soybean oil (SME) used in this study were SoyGold obtained from Ag Environmental Products (Lenexa, KS).

**Carbonate preparation.** The carbonates used in this investigation were prepared in accordance with the literature by Kenar *et al.* (16).

**Refractive index and density.** The refractive indices were determined on an Abbe 3T refractometer (Attago, Inc., Bellevue, WA) held at a constant temperature with a constant-temperature water bath. Density was determined by precisely measuring the weight of desired carbonate in a 1-mL volumetric flask calibrated for the same volume of water at the specified temperature. The densities were determined in duplicate.

**Viscosity.** Kinematic viscosity of the carbonates was determined in accordance with American Society for Testing and Materials (ASTM) method D445 (17). Samples were run in triplicate (SD 0.00–0.03 mm<sup>2</sup>/s) and mean viscosities determined.

**CN.** CN of seven carbonates were determined in an Ignition Quality Tester<sup>TM</sup> (IQT) as described previously (13). That the results of the CN determination by the IQT were reproducible was additionally checked by determining the CN of methyl oleate. In the present series of experiments, the CN of methyl oleate was determined as 58.3. This compares well with the results from previous work in which the CN of methyl oleate was determined as 59.3 (13).

**CP and PP.** CP and PP were measured in accordance with ASTM methods D5773 (18) and D5949 (19) using a model PSA-70S automatic analyzer from Phase Technology (Richmond, British Columbia, Canada). This analyzer requires ~150 µL per sample measurement and yields both CP and PP measurements within 6–12 min. The sample is cooled at 1.5 ± 0.1°C/min by a Peltier device while continuously being illuminated and monitored by an array of optical detectors. The temperature at which a cloud of wax crystals is first detected is recorded as the CP (SD = 0.058–0.12). This technique for measuring CP of petroleum products demonstrates little bias in comparison with “manual” methods such as ASTM method D

2500 (18,20). The analyzer measures PP by cooling the sample and applying a moving force in the form of a pressurized pulse of N<sub>2</sub> gas at constant temperature intervals. The lowest temperature at which movement of the surface is detected on application of force is recorded as PP (SD 0.0–1.2). For this work, samples were tested for surface movement in temperature intervals of 2°C.

**Lubricity.** The lubricity of the carbonates was investigated by the method ASTM D6079, high-frequency reciprocating rig (HFRR; PCS Instruments, London, England) (21). All samples were run in duplicate. Wear scar values for neat low-lubricity ultra-low sulfur diesel (ULSD) fuel are 613 and 617 microns (µm) at 25°C and 651 and 636 µm at 60°C (22), well above the wear scar value of 460 or 520 µm prescribed in standards (European standard EN 590 and ASTM D975, respectively) for petrodiesel fuel. The film and (coefficient of) friction values recorded by the HFRR software are also included in Table 3.

**Surface tension.** Surface tension was determined using the ring method on a Krüss (Charlotte, NC) digital tensiometer K10T as described previously (23).

## RESULTS AND DISCUSSION

The various physical properties of carbonate compounds such as density, refractive index, m.p., b.p., viscosity, and CN are reported in Table 1. For the linear carbonates, as the alkyl chain length increases there is an increase in m.p., as expected for the series dioctyl (–18.0°C), dinonyl (–3.0°C), and didecyl (3.0°C) carbonate. Incorporation of two *cis* double bonds in the linear dioleoyl carbonate structure effectively lowered its m.p. relative to didecyl carbonate even though dioleoyl carbonate has 16 additional carbon atoms. Introduction of branching in the carbonate compounds decreased their m.p. dramatically. All the branched carbonates, bis(2-ethylhexyl), bis(2-butyloctyl), and bis(2-hexyldecyl) carbonate, remained liquid at temperatures below –50°C.

Densities and refractive indices of the carbonate compounds are also reported in Table 1. As can be seen, the density of the straight-chain carbonates decreases slightly as the number of carbon atoms increases. This same general trend was observed

**TABLE 1**  
Physical Properties of Oleochemical Carbonates

Compound	Formula	Density		Refractive index n <sub>D</sub> <sup>c</sup>	m.p. (°C)	b.p. <sup>b</sup> (°C)	Kinematic viscosity <sup>c</sup> (mm <sup>2</sup> /s)	Cetane number (CN)
		M.W.	D <sup>a</sup> (g cm <sup>-3</sup> )					
Dioctyl carbonate	C <sub>17</sub> H <sub>34</sub> O <sub>3</sub>	286.450	0.895	1.4360 <sup>20.2</sup>	–18	100 <sup>0.37</sup>	4.94	63.9
Dinonyl carbonate	C <sub>19</sub> H <sub>38</sub> O <sub>3</sub>	314.503	0.887	1.4392 <sup>20.2</sup>	–3	125 <sup>0.48</sup>	6.11	70.4
Didecyl carbonate	C <sub>21</sub> H <sub>42</sub> O <sub>3</sub>	342.556	0.884	1.4422 <sup>20.2</sup>	3	142 <sup>0.14</sup>	8.10	82.5
Dioleoyl carbonate	C <sub>37</sub> H <sub>72</sub> O <sub>3</sub>	562.950	0.879	1.4628 <sup>20.3</sup>	0	195 <sup>0.48</sup>	19.6	107.2
Bis(2-ethylhexyl) carbonate	C <sub>17</sub> H <sub>34</sub> O <sub>3</sub>	286.450	0.897	1.4368 <sup>20.4</sup>	<–50	100 <sup>0.50</sup>	4.36	47.2
Bis(2-butyloctyl) carbonate	C <sub>25</sub> H <sub>50</sub> O <sub>3</sub>	398.663	0.884	1.4464 <sup>20.3</sup>	<–50	140 <sup>0.28</sup>	12.1	74.2
Bis(2-hexyldecyl) carbonate	C <sub>33</sub> H <sub>66</sub> O <sub>3</sub>	510.875	0.870	1.4522 <sup>20.4</sup>	<–50	185 <sup>0.38</sup>	22.1	101.7

<sup>a</sup>Density measured at 22°C.

<sup>b</sup>Superscript denotes pressure in Torr.

<sup>c</sup>Kinematic viscosity at 40°C.

for the branched carbonate compounds. Comparison of dioctyl carbonate with bis(2-ethylhexyl) carbonate, which both have the same molecular formula, shows the densities to be similar at 0.895 and 0.897, respectively. The refractive indices of the straight-chain and branched compounds both increased as the number of carbon atoms increased. Comparison of the refractive indices of either dioctyl or 2-ethylhexyl carbonate with that of methyl palmitate (1.4510 at 20°C), which has the same number of carbon atoms, shows that the alkyl ester has a significantly higher refractive index.

**Kinematic viscosity.** The data for the kinematic viscosity of the carbonates was determined analogously to that of various fatty compounds and is shown in Table 1. As ascertained for the fatty compounds (24), the viscosity of the carbonates increased with increasing number of carbon atoms. The additional oxygen contained in carbonate compounds relative to alkyl esters compounds containing the same number of carbons moderately increased viscosity as exemplified by comparison of the kinematic viscosities of dioctyl and dinonyl carbonate to the kinematic viscosities of methyl palmitate (4.32 mm<sup>2</sup>/s) and methyl stearate (5.61 mm<sup>2</sup>/s), respectively (25). In contrast, branching reduces viscosity modestly, as can be seen by comparison of the data for bis(2-ethylhexyl) carbonate and dioctyl carbonate, which have the same number of carbon atoms. A minor effect of branching on viscosity was reported for branched alkyl esters vs. straight-chain alkyl esters (26). The effect of branching on viscosity depends likely on the chain lengths of the branched hydrocarbon moiety.

**CN.** The CN is a major fuel quality index of diesel fuels. The CN is a dimensionless descriptor for the ignition delay time a fuel experiences on injection into the cylinder of a diesel engine. It is therefore related to the combustion of a diesel fuel and, ultimately, exhaust emissions. For example, it has been shown that higher CN of a fuel can result in reductions in the exhaust emissions of nitrogen oxides (NO<sub>x</sub>) (27).

To ascertain that the present compounds would not compromise combustion properties when used as a diesel fuel component and for the sake of comparison with alkyl esters, the CN of seven carbonates were determined in an IQT. The IQT is an alternative to the complex and cumbersome CN determination by the standard ASTM D613 and forms the basis of the new standard ASTM D6890. It is faster and uses considerably less test material while giving results of comparable reproducibility and repeatability. The IQT has previously been applied to the CN determination of straight-chain and branched fatty esters (13). The results of the CN determinations are given in Table 1.

The results show that the CN of the carbonates increased with increasing chain length. A comparison of the CN of dioctyl carbonate and bis(2-ethylhexyl)carbonate, which have the same number of carbon atoms, showed that branching reduces CN. Both of these trends have been described or predicted for the esters of FA (13,28, and references therein). The results also showed that interrupting the chain of methylenes reduces CN. The CN of methyl palmitate by the IQT method is 85.9 (13) whereas that of dioctyl carbonate, which has the same number of carbon atoms, is 63.9 (Table 1). The additional

ether-like oxygen atom present in the carbonate moiety relative to the ester group does not appear to enhance CN between these two functionalities; rather the uninterrupted carbon chain appears to be the decisive factor. Similarly, the CN of dinonyl carbonate is lower than that of methyl stearate, for which CN = 101 by the IQT method (13). Even the CN of didecyl carbonate, which possesses more carbon atoms than either methyl palmitate or methyl stearate, is still lower than the CN of these methyl esters. Furthermore, the high CN of dioleoyl carbonates shows that the unsaturation present in the carbon chain reduces CN less than interruption of the alkyl chain by a carbonate group. The results show that carbonates are useful for further assessment and confirmation of structural effects on combustion compared with FA alkyl esters. However, the CN values of all carbonates investigated here are well above the minimum CN of 40 prescribed for conventional petroleum-derived diesel fuel in the standard ASTM D975. They also exceed the minimum CN of 47 called for in the standard ASTM D6751 developed for biodiesel fuel. The results show the present compounds would not compromise diesel fuel combustion, and, to the contrary, may provide an increase in CN depending on their concentration and the CN of the base fuel.

**Cold flow properties.** Because the branched carbonates bis-2-ethylhexyl, bis-2-butyloctyl, and bis-2-hexyldecyl carbonate had promising low-temperature properties along with acceptable CN, we studied their effects on biodiesel cold flow properties. Table 2 summarizes data showing the effects on cold flow properties when each of the three carbonate compounds is added to biodiesel. At an additive concentration (loading = 1000 ppm, 0.1 wt%), bis-2-ethylhexyl carbonate did not improve CP whereas bis-2-hexyldecyl and bis-2-butyloctyl carbonates increased CP by only ~0.5°C ( $P < 0.01$ ) relative to untreated SME. Hence, effects of all three carbonates on CP appear to be minimal. Although increasing carbonate concentration to 10,000 ppm (1 wt%) generally resulted in decreases in CP, net decreases were also minimal (<1°C). Although PP tended to decrease slightly for all three additives, decreases did not exceed experimental resolution (sample temperature intervals = 2°C) used in this study.

**Lubricity.** Table 3 shows the HFRR lubricity data obtained for the neat carbonates and when blended with low-lubricity ULSD. The lubricity of the neat carbonates is within the acceptable limits of HFRR with a tendency toward improved lubricity as the number of carbon atoms in the alkyl chain increases. However, at a 1% blend level with low-lubricity ULSD, none of the selected carbonates imparted acceptable lubricity by HFRR. This result correlates with recent results (22) in which

**TABLE 2**  
Cold Flow Properties of SME Treated with Carbonates<sup>a</sup>

Carbonate	Concentration (ppm)	CP (°C)	PP (°C)
None	0	5.4	6 <sup>b</sup>
Bis(2-ethylhexyl) carbonate	1000	5.53	5
Bis(2-butyloctyl) carbonate	1000	5.93	5
Bis(2-hexyldecyl) carbonate	1000	5.9	5

<sup>a</sup>SME, FAME of soybean oil (SoyGold); CP, cloud point; PP, pour point.

<sup>b</sup>Variance = 0.



**TABLE 3**  
**High-Frequency Reciprocating Rig Data (HFRR; wear scar in  $\mu\text{m}$ ) of Carbonates**

Material	Wear scar ( $\mu\text{m}$ ) <sup>a</sup>						Film (%)		Friction	
	25°C			60°C			25°C	60°C	25°C	60°C
	X	Y	Avg.	X	Y	Avg.				
Dioctyl carbonate	226; 268	190; 244	208; 256	381; 398	368; 394	<i>375; 396</i>	93; 74	93; 71	0.112; 0.112	0.171; 0.216
Dinonyl carbonate	180; 189	177; 175	179; 182	425; 436	396; 392	<i>411; 414</i>	100; 100	60; 61	0.088; 0.095	0.200; 0.210
Didecyl carbonate	172; 192	156; 173	164; 183	379; 402	365; 383	<i>372; 393</i>	99; 99	76; 73	0.072; 0.081	0.160; 0.165
Hexadecyl carbonate	ND <sup>b</sup>	ND <sup>b</sup>	ND <sup>b</sup>	301; 330	212; 243	<i>257; 287</i>	ND <sup>b</sup>	95; 93	ND <sup>b</sup>	0.105; 0.106
Dioleyl carbonate	172; 175	165; 115	169; 145	323; 308	302; 290	<i>313; 299</i>	98; 98	86; 89	0.092; 0.088	0.119; 0.121
Bis(2-butyloctyl) carbonate	141; 135	111; 105	126; 120	322; 328	325; 313	<i>324; 321</i>	100; 100	84; 84	0.111; 0.103	0.157; 0.152
Bis(2-hexyldecyl) carbonate	142; 143	110; 131	126; 137	331; 335	314; 316	<i>323; 326</i>	100; 100	76; 74	0.094; 0.088	0.157; 0.162
Carbonates as fuel additives										
1% Hexadecyl carbonate in ULSD w/o additives <sup>c</sup>	ND	ND	ND	634; 671	612; 618	<i>623; 645</i>	ND	12; 10	ND	0.337; 0.367
1% Dioleyl carbonate in ULSD w/o additives	ND	ND	ND	622; 618	584; 563	<i>626; 666</i>	ND	13; 10	ND	0.332; 0.338

<sup>a</sup>Values for X and Y are the maximum values (microns =  $\mu\text{m}$ ) for the dimensions of the wear scar on the steel ball used in the HFRR test. The X and Y values are then averaged to the values given in the third column for each temperature. The italicized average values for 60°C are those that should be compared with the limits prescribed in petrodiesel standards.

<sup>b</sup>Not determined since hexadecyl carbonate is a solid at 25°C.

<sup>c</sup>ULSD, ultra-low sulfur diesel.

polar groups such as  $-\text{COOH}$  or  $-\text{OH}$  or terminal  $-\text{HC}=\text{O}$  (aldehydes) were found to be more effective in imparting lubricity than neat methyl esters. The additional oxygen present in the carbonate moiety by itself does not enhance lubricity by HFRR compared with methyl esters (22). In the case of neat methyl esters, the carbonyl moiety is responsible for lubricity enhancement whereas the alcohol portion of the ester group has little or no influence (22). This general observation appears to hold for carbonates too, and the lubricity performance of neat carbonates is in all likelihood also attributable to the carbonyl moiety of the carbonate group.

**Surface tension.** Two carbonates, a branched and a straight-chain sample, were selected for studying the effect of carbonates on the surface tension of a 1 N NaOH solution. Neither of the selected carbonates, didecyl carbonate and bis-2-butyloctyl carbonate, lowered the surface tension of the 1 N NaOH solution (blank value 74.3 mN/m) significantly. The surface tension was lowered to 57.5 mN/m with didecyl carbonate at a concentration of  $7.5 \times 10^{-4}$  and 61.5 mN/m with bis-2-butyloctyl carbonate at a concentration of  $2.5 \times 10^{-3}$ .

The present results show that carbonates possess properties resembling those of FA alkyl esters with similar trends based on compound structure. The CN, kinematic viscosities at 40°C, and m.p. of the carbonates studied ranged from 47–107, 4.9–22.6  $\text{mm}^2/\text{s}$ , and +3 to below  $-50^\circ\text{C}$ , respectively, depending on carbon chain length and branching. The carbonate CN are lower than those for FA alkyl esters containing the same number of carbons, owing to interruption of the  $\text{CH}_2$  chain by the carbonate moiety. The neat carbonates had acceptable lubricity, as determined by HFRR, that improved as chain length increased. The addition of carbonate compounds as additives in biodiesel was not able to significantly improve cold flow

properties as hoped at concentrations up to 10,000 ppm (1 wt%). However, because of their reduced potential for corrosiveness upon degradation, they are attractive candidate compounds for industrial applications.

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