Research Article

Synthesis and Characterization of an Iron-Containing Fatty Acid-Based Ionomer

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One of the desirable research goals today is to convert agro-based raw materials into low-cost functional polymers. Among the readily available natural raw materials are the fatty acids that can be obtained from the hydrolysis of plant oils or from the paper industry as byproducts. In this work, a novel iron-containing ionomer has been prepared through the reaction of fatty acids with steel dust or iron powder in the presence of carbon dioxide and water. Characterization has been achieved via 1H and 13C NMR, FT-IR, and size exclusion chromatography. The product has been shown to have an ionomeric structure, consisting of oligomers of fatty acid carboxylates (derived from Diels-Alder reaction) coupled with iron(II) and iron(III) ions (from the oxidation of iron). Because the fatty acid oligomers have low molecular weights, the ionomer easily dissolves in a solvent and can be made into different physical forms, such as liquid, solid, film, or foam.

1. Introduction

There is an intense interest in finding alternative sources of raw materials that are derived from renewable, sustainable sources rather than petroleum and other fossil fuels [1, 2]. Furthermore, in view of the recent concern about the health and environmental impact of many chemicals, “green chemistry” (which involves the design of chemical products and processes that reduce or eliminate the use or generation of hazardous substances) is increasingly being adopted in research and development [3, 4]. One of the green chemistry approaches is to use agro-based plant oils or animal fats as raw materials for bio-based products [5–7]. The major benefits of oils and fats are their relatively low cost, commercial availability, and their nontoxic and biodegradable properties. In recent years, a large number of papers have been published on the syntheses of polymers from oils and fats [8, 9]. Some examples are the heat-induced polymerization of oils [10–13], cationic polymerization [14–18], and polymer formation after the pericyclic reaction of a triglyceride with a selected amine [19, 20]. Moreover, an acrylate can be grafted onto a triglyceride and subsequently polymerized [21, 22]. The triglyceride can also be modified to bear a hydroxy group, which is then reacted with di-isocyanates to form a polyurethane [6, 8, 9]. Finally, the double bonds in triglycerides can be converted into epoxy groups, which can then be polymerized [23, 24]. Efforts have also been made to polymerize epoxidized fatty acid methyl ester [25]. Most of these publications involve triglycerides or their methyl esters. Thus far, there has been relatively little effort in converting free fatty acids into polymers.

We have been attracted to tall oil fatty acids (TOFA, CAS# 61790-12-3) because they are inexpensive and available. The term tall oil [26] comes from the Swedish word tallol, meaning pine tree oil, and is a byproduct of the Kraft papermaking process. It comprises approximately 48% oleic acid, 46% linoleic acid and isomers, 3% palmitic and stearic acids, and 3% other fatty acids [27]. The stimulus for this work was the discovery [28] that a mixture of TOFA, metallic iron, carbon dioxide, and trace water can be
transformed into a solid polymer. The goal of this work was to carry out more detailed studies of the TOFA/iron/CO₂/water system in order to elucidate the reaction mechanism of this novel polymer reaction. In particular, the role of each of these components (fatty acids, iron, carbon dioxide, and water) was clarified. Although the emphasis of this work was on TOFA, similar reactions can be carried out with fatty acids derived from soybean oil, cottonseed oil, and other plant oils.

2. Materials and Methods

2.1. Materials. The TOFA used in this study was manufactured by Kraton Corporation (Houston, TX, USA) with the product name SYLFAT™ FA1 and was acquired through a global plastic and chemical distributor, Nexeo Solutions Inc. (The Woodlands, TX, USA). Steel dust was obtained as a residue from the shot blasting operation of Schuff Steel (Phoenix, AZ, USA), which uses steel abrasives manufactured by Ervin Amasteel (Adrian, MI, USA). The steel wool was obtained from Global Material Technologies (Chicago, IL, USA). Electrolytic and hydrogen-reduced iron powder was obtained from Höganas Corporation (Hollisopple, PA, USA). Carbonated water was taken from Perrier “sparkling natural mineral water,” purchased from a local Walmart store at Peoria, IL, USA.

2.2. Synthetic Procedure. Two procedures were employed for this reaction. In procedure A, TOFA and iron powder were mixed in a volume ratio of 1:0.5 to 1:1 and placed inside a glove box filled with CO₂. A small amount of water was added up to 1% by oil volume. The mixture thickened in the course of time (ranging from 1 hour to overnight).

Procedure B involved 1 part by weight of TOFA, 0.5 part steel dust or iron powder, and up to 0.025 part carbonated water. These were mixed together at 35 to 45°C. Some bubbling occurred within a few minutes, which gradually subsided in about one hour while the mixture thickened and eventually hardened.

2.3. Characterization. The TOFA products were analyzed without further treatments. For NMR analysis, each TOFA-iron sample was dissolved at about 10% concentration in chloroform-d in a NMR tube. The ¹H and ¹³C NMR spectra were acquired on a Bruker DRX 400 spectrometer (Karlsruhe, Germany) at ambient temperature using standard operating conditions. The ¹H and ¹³C chemical shifts were referenced to tetramethylsilane at 0 ppm. Spectral assignments were made using previously reported chemical shifts of related compounds [29], empirical chemical shift rules [30], and automated shift prediction software [31].

FT-IR spectra were obtained on a Nicolet iS10 FT-IR spectrometer (Thermo Fisher Scientific Inc., Waltham, MA, USA) equipped with a Smart Orbit single-bounce ATR accessory with a diamond crystal. For each spectrum, 32 scans were collected at room temperature at a spectral resolution of 4 cm⁻¹ between 600 and 4000 cm⁻¹ using a DTGS detector and KBr beam splitter. Data were processed with the OMNIC software (version 9.2.98).

Size exclusion chromatography (SEC) profiles were obtained on a HPLC system including a 1515 isocratic HPLC pump, 717plus automated injector, column heater, and Breeze software obtained from Waters Corporation (Milford, MA, USA). Columns used for separation were a pair of PLgel 3µm MIXED-E, 300×7.5 mm and a PLgel 5 µm Guard, 50×7.5 mm (part numbers PL1110-6300 and PL1110-1520, respectively) from Polymer Laboratories Ltd. (Varian Inc., Amherst, MA, USA). Signals generated from an Optilab rEX refractive index detector obtained from Wyatt Technology Corporation (Santa Barbara, CA, USA) were processed using ASTRA V software also from Wyatt Technology Corporation. THF was used as the mobile phase at a flow rate of 1 mL/min and columns were maintained at 40°C. The samples were dissolved in THF at about 0.1% concentration, stabilized with butylated hydroxytoluene from Thermo Fisher Scientific (Suwanee, GA, USA) at a concentration of about 4.00 × 10⁻³ g/mL. The Waters autosampler was used to make 100 μL injections from a 1 mL sample vial. Calibration of molecular weights was carried out with linear polystyrene standards (Polymer Laboratories, Santa Clara, CA, USA, Mn = 580-100 K, Mw/Mn = 1). Astra V software was used to calculate molecular weight and to export chromatographic data to Microsoft Excel (Redmond, WA, USA).

3. Results

In this novel reaction, four components were mixed together: fatty acids, metallic iron, water, and CO₂. Within hours, the organometallic composite darkened, thickened, and (in some cases) solidified. A flammable gas byproduct was also produced. Many samples were made, using either of the two procedures given in Section 2. In particular, seven samples (Table 1) were produced via these procedures in order to facilitate the characterization of the reaction products.

The SEC molecular weight information of these samples is shown in Table 1. The samples were all dissolved at 0.1% in THF; thus, the interactions between the polymer and iron cations that might be present in the samples would be minimized. Since oleic and linoleic acids have Mw of 282 and 280 g/mol, respectively, all reaction products (with Mw between 914 and 2420 g/mol) were oligomers. Samples A1-A3 were the products made according to procedure A. Sample A1 seemed to be heterogeneous, with a mixture of unreacted fatty acids and reacted oligomers, giving a large polydispersity of 4.8. As more iron and CO₂ were added in samples A2 and A3, both Mn and Mw increased, indicating increasing polymerization of all the fatty acids. Samples B1-B3 were the reaction products obtained from the use of procedure B. Sample B1 seemed to be heterogeneous, showing the presence of both unreacted fatty acids and reacted oligomers. With the addition of more carbonated water in sample B2, both Mn and Mw increased, suggesting that most of the fatty acids participated in the polymerization. Sample B3 was made with TOFA, iron, carbonated water, and CO₂. The coaddition of CO₂ probably promoted the molecular weight buildup of reacted fatty acids, and a larger polydispersity (5.6) was observed; the possible reaction mechanism will
be described in Discussion. Sample C1 was the reaction product from linoleic acid, which had also been exposed to air. This sample had the highest molecular weight (Mn 1400 and Mw 2420) among all the samples in Table 1.

The FT-IR spectra for the starting TOFA (sample R) and the seven reaction products (as they were made, without solvent) are given in Figure 1. The spectrum for sample R was compatible with those of fatty acids [32, 33]. Particularly distinctive was the 1710 cm\(^{-1}\) peak, corresponding to the C=O stretching of the carboxylic acid; in acid salts, the C=O asymmetric stretching peak was reported to appear at around 1000 cm\(^{-1}\); the spectral pattern was somewhat similar to the IR spectrum of oleic acid-coated cobalt nanoparticles [35]. The FT-IR spectra of samples B1-B3 also showed the presence of fatty acids and fatty acid salts. Moreover, in the 13C NMR spectra, the peak for the carboxylate (COO\(^-\)) appears noticeable modulated by the presence of a mixture of fatty acids and fatty acid salts at different proportions. In the spectrum for sample A3, there were additional broad peaks at around 1000 cm\(^{-1}\); the spectral pattern was somewhat similar to the IR spectrum of oleic acid-coated cobalt nanoparticles [35]. The FT-IR spectra of samples B1-B3 also showed the presence of fatty acids and acid salts; the peaks for acid salts were strong in sample B1 but weak in samples B2 and B3. Moreover, in the 1400-1600 cm\(^{-1}\) region for samples A1, A2, A3, and B1, there appears noticeable modifications of the modes of vibrations attributed to the carboxylate group of fatty acid ion salts, e.g., symmetric stretching vibration between 1380 and 1480 cm\(^{-1}\) and asymmetric stretching vibration between 1480 and 1600 cm\(^{-1}\) [32–34]. In the 500-800 cm\(^{-1}\) region for samples B1, B2, and B3, the intensity of the metal-O absorption band (adjacent to the (CH\(_2\))\(_n\) rocking peak at 722 cm\(^{-1}\)) is strong [36, 37]. However, for samples A1, A2, and A3, the delocalized absorption bands at 550-670 cm\(^{-1}\) seem to coexist with and are more preponderant than the metal-O absorption band.

The \(^{13}\)C NMR spectra of the starting TOFA and the seven reaction products, dissolved in chloroform-d, are given in Figure 2. The spectrum for sample R showed the presence of oleic and linoleic acids. The assignments [29–31] are noted in the spectrum. For sample A1 (the reaction product of TOFA made with procedure A), the amount of oleic acid stayed about the same, but linoleic acid peaks noticeably decreased in intensity. The peaks for carboxylate (COO\(^-\), ca. 180 ppm), C2 (34 ppm), and C3 (24.7 ppm) all decreased in intensity such that they were barely observable. Since the IR spectra clearly demonstrated the conversion of fatty acid to carboxylate acid salt in the reacted samples, this intensity reduction must be due to the oxidation of iron to Fe(II) and Fe(III) cations, which then became the counterions for the carboxylate ions of the fatty acid salt (both oleate and linolate). As Fe(II) and Fe(III) were paramagnetic, the \(^{13}\)C peaks for the carboxylate, C2, and C3 (which were located near the Fe cation) underwent rapid electron relaxation and were broadened beyond detection in the NMR spectra. The intensities of the carbons further away from iron were progressively less affected.

For sample A2, broad peaks were observed for the entire \(^{13}\)C NMR spectrum because of the large amount of Fe(II) or Fe(III) present and (to a lesser extent) the formation of

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sample description</th>
<th>Proc.</th>
<th>NMR analysis(^a)</th>
<th>FT-IR analysis</th>
<th>Mn</th>
<th>Mw</th>
<th>DP</th>
</tr>
</thead>
<tbody>
<tr>
<td>R</td>
<td>TOFA</td>
<td>None</td>
<td>Oleic and linoleic fatty acids</td>
<td>Fatty acid</td>
<td>281(^b)</td>
<td>281(^b)</td>
<td>1</td>
</tr>
<tr>
<td>A1</td>
<td>TOFA : iron : water–1 : 0.5 : 0.01 (CO(_2) present)</td>
<td>A</td>
<td>Oleic (st), linoleic (md), carboxylate (vw)</td>
<td>Fatty acid, acid carboxylate</td>
<td>280</td>
<td>1320</td>
<td>4.8</td>
</tr>
<tr>
<td>A2</td>
<td>TOFA : iron : water–1 : 0.8 : 0.01 (CO(_2) present)</td>
<td>A</td>
<td>Oleic (st), linoleic (md), carboxylate (0), peaks broadened</td>
<td>Fatty acid, acid carboxylate</td>
<td>450</td>
<td>1250</td>
<td>2.8</td>
</tr>
<tr>
<td>A3</td>
<td>TOFA : iron : water–1 : 1 : 0.01 (more CO(_2) present)</td>
<td>A</td>
<td>Oleic (wk), linoleic (vw), carboxylate (0), peaks broadened even more</td>
<td>Fatty acid, acid carboxylate, metal-carboxylate complex</td>
<td>1090</td>
<td>1580</td>
<td>1.4</td>
</tr>
<tr>
<td>B1</td>
<td>TOFA : iron : carbonated water–1 : 0.5 : 0.015</td>
<td>B</td>
<td>Oleic (st), linoleic (md), carboxylate (0), peaks slightly broadened</td>
<td>Fatty acid, trace carboxylate, metal-O bonding</td>
<td>284</td>
<td>914</td>
<td>3.2</td>
</tr>
<tr>
<td>B2</td>
<td>TOFA : iron : carbonated water–1 : 0.5 : 0.025</td>
<td>B</td>
<td>Oleic (st), linoleic (md), carboxylate (vw), peaks broadened</td>
<td>Fatty acid, trace carboxylate, metal-O bonding</td>
<td>875</td>
<td>1100</td>
<td>1.3</td>
</tr>
<tr>
<td>B3</td>
<td>TOFA : iron : carbonated water–1 : 0.5 : 0.025 (CO(_2) also bubbled into a mixture)</td>
<td>B</td>
<td>Oleic (st), linoleic (md), carboxylate (w), peaks broadened</td>
<td>Fatty acid, trace carboxylate, metal-O bonding</td>
<td>340</td>
<td>1870</td>
<td>5.6</td>
</tr>
<tr>
<td>C1</td>
<td>Linoleic acid : iron : water–1 : 0.5 : 0.01 (CO(_2) and air present)</td>
<td>A</td>
<td>Oleic (st), carboxylate (st), some conjugations present due to oxidation</td>
<td>Fatty acid, some metal-carboxylate complexes</td>
<td>1400</td>
<td>2420</td>
<td>1.8</td>
</tr>
</tbody>
</table>

\(^{a}\)Relative abundance of oleic and linoleic acids and carboxylate was estimated as strong (st), medium (md), weak (wk), very weak (vw), and absent (0) from peak heights in the \(^{13}\)C NMR spectra. \(^{b}\)This is the reference sample; only oleic and linoleic acids are assumed present, with molecular weights of 282 and 280, respectively.
higher molecular weight species. In this case, the carboxylate, C2, and C3 peaks were completely broadened beyond detection in the spectrum. The spectrum for sample A3 exhibited even broader peaks. In the olefin region, only a small amount of oleic acid was left. Most of the linoleate olefin peaks (at positions 9, 10, 12, and 13) were gone, and the linoleate C11 (at 25.8 ppm) only showed a very small peak. The carboxylate peak was too broad to be visible. Samples B1-B3 (the reaction products obtained from procedure B) gave similarly looking spectra. The carboxylate, C2, and C3 peaks were either much diminished, broadened, or disappeared due to binding with Fe(II) or Fe(III) cations. The line broadening of the peaks increased slightly from B1 to B3, probably due to the molecular weight increase of the oligomers formed. Sample C1 (the reaction product of linoleic acid) showed a small amount of conjugated linoleic acid structures due to oxidation that can be seen as small peaks at the 127-136 ppm region; these structures can be more clearly observable. All the peaks in samples B1-B3 were somewhat diminished, broadened, or disappeared due to binding with Fe(II) or Fe(III) cations. In samples A2 and A3, the spectra were broad due to the high levels of Fe(II) and Fe(III) cations and carboxylate binding such that C2 and C3 methylenes were completely broadened beyond detection. In samples B2 and B3, the C2 and C3 methylenes were small but still observable. All the peaks in samples B1-B3 were somewhat broad, although not as broad as samples A2 and A3.

A note may be made of the 1H NMR spectrum of sample C1, which was the only sample exposed to air. For clarity, a spectrum with higher magnification is shown in Figure 4. Additional small peaks in the 4.3-6.6 ppm region (marked with asterisks) were due to the oxidation of diolefin in linoleate to form a conjugated structure adjacent to a hydroperoxide:

\[
\text{RCOO}^- + \text{OH} \rightarrow \text{RCOOH}
\]

The observed 1H spectrum (Figure 4) matched very well with the previously published spectra of oxidized linoleic moiety [38, 39]. Accordingly, the 4.35 ppm peak was due to the methine next to hydroperoxy functionality and the peaks at 5.3-6.6 ppm corresponded to various conjugated diene structures. By the way, in the 13C NMR spectrum the 13C peak next to hydroperoxy can be found at 86.5 ppm, which is not shown in Figure 2.

4. Discussion

In view of the results from 13C NMR, 1H NMR, FT-IR, and size exclusion chromatography, a tentative reaction mechanism can be formulated. It is clear from NMR and IR data that during the reaction iron is oxidized to Fe(II) or Fe(III) cations, which then serve as counterions to the fatty acid carboxylate. Thus, one of the initial reactions involves the reaction of iron with the fatty acid to form the carboxylate salts between Fe(II) and Fe(III) cations (equation 2a):

\[
3 \text{RCOOH} + \text{Fe} \rightarrow (\text{RCOO}^-)_3\text{Fe}^{3+} + 3/2\text{H}_2
\]  

\[
2 \text{RCOOH} + \text{Fe} \rightarrow (\text{RCOO}^-)_2\text{Fe}^{2+} + \text{H}_2
\]

This is not a surprising result as the carboxylic acid moiety is known to be reactive. A similar reaction in a different context has been previously reported [40]. Note that one Fe(II) cation can bind with two fatty acid salts and one Fe(III) cation with three fatty acid salts. In addition, some conversion of the linoleic acid to conjugated linoleic acid is possible due to Fe-induced isomerization [19, 20] (and possibly hydroperoxide formation [38, 39, 41]), leading to the Diels-Alder reaction between the fatty acids that couples the two fatty acids together:

\[
2 \text{RCOO}^- \rightarrow \text{“OCOR – RCOO”}
\]

When repeated, equation 3 can generate oligomers from the conjugated fatty acids (equation 4), as we indeed observed in the SEC data:

\[
\text{“OCOR–RCOO”} \rightarrow \text{“OCOR–(R) n–RCOO”}
\]

Equation 2a also generates hydrogen. It is noteworthy that during this initial reaction stage a flammable gas (probably hydrogen) was found in the form of bubbles.
throughout the oil-iron mixture and caused it to expand and foam up. When the froth of bubbles was ignited, a wave of flame spread across the surface. One bursting bubble could ignite the others around it. Thus, hydrogen is expected and was indeed observed. In addition, it is also possible that the iron complexes may catalyze some dehydrogenation of the fatty acids, as suggested by a paper in the literature [42]. This dehydrogenation of the fatty acids results in the formation of more olefins, which leads to more of the Diels-Alder reactions. Through the combination of equations (2a), (3), and (4), an ionomer is produced, consisting of Fe(II) and Fe(III) binding with the carboxylates and Diels-Alder coupling products. A possible reaction scheme is shown below for Fe$$^{++}$$:

$$\text{COO}^- \cdot \text{OCOR}^- \cdot \text{Fe}^{++} \rightarrow \left[\text{Fe}^{++} \cdot \text{OCOR}^- \cdot \text{Fe}^{++} \right]_{n}$$

where the subscripts $$n$$ and $$m$$ are integers. For simplicity, the structure on the right in equation 5 shows only Fe$$^{++}$$/-carboxylate binding for the chain ends of the fatty acid oligomers. In reality, Fe(II) and Fe(III) ions bind randomly with all available carboxylates. This general picture is confirmed by NMR and FT-IR data, where both the carboxylate salt structure and the Fe cation/carboxylate binding were observed.

Equations (2a), (3), (4), and (5) rationalize the formation of the ionomer, which causes the visible increase in the viscosity of the reaction products. A further detail relates to carbon dioxide and water that promote the reaction. As we searched the literature, we found out that the corrosion of iron or mild steel is promoted by CO$$^2$$ and water [43–47]. Thus, carbon dioxide presumably reacts with water to form carbonic acid (H$$^2$$CO$$^3$$), and iron is solubilized and oxidized as a result. The major iron products seem to be FeCO$$^3$$ and iron oxides [43, 45]. Carbonic acid is considered to be a major factor in the corrosion of steel, and hydrogen evolution has been observed in electrochemical
studies [46, 47]. Although the overall reactions are complex, a simplified reaction [45] can be shown as follows:

\[ \text{CO}_2 + \text{H}_2\text{O} + \text{Fe} \rightarrow \text{FeCO}_3 + \text{H}_2 \]  

Thus, iron seems to play multiple roles in our reaction system: it reacts with carbonic acid to initiate the reaction process, with fatty acid to form iron carboxylate, and with the linoleic functionality to form oxidized conjugated linoleic acid and it possibly dehydrogenates some fatty acids to produce more olefins.

From the current investigation, it appears that the outline of the reaction pathways leading to ionomer formation is mostly clear. However, some of these pathways are rather complex, and further work is needed to clarify the nuances of these reactions. The possible role of air in the reaction pathways will also need to be explored further.

It may be noted that the ionomeric products were similar when different sources of iron were used (steel powder or steel wool). In a previous work by Das et al. [48], the same steel powder was used (and at comparable amounts by volume) for the formation of an iron carbonate composite. Although the final products of the current work and the earlier Das paper were different, the Das paper illustrated another utility of iron carbonation. Moreover, in the paper of Das et al. [48], additional information was given on the composition and particle size of the steel powder and the porosity and other characteristics of the iron carbonate complex.

As for applications, this novel fatty acid-based ionomer can be used as a coating for construction, as a roofing compound, and as a sealant. An example of its application as a coating for adobe is shown in Figure 5, where the iron-containing ionomer serves as an outer weather-resistant layer. In the literature [49, 50], ionomers are also known for a variety of other uses including packaging, fuel cell membranes, electrochemical synthesis, catalysis, shape memory and self-healing materials, and supramolecular polymer systems. Some of these uses may or may not be probable for this material, and further exploration would be needed to determine the scope of future applications.

\[ \text{R} \]
\[ \text{CH}=\text{CH} \]
\[ \text{A1} \]
\[ \text{A2} \]
\[ \text{A3} \]
\[ \text{B1} \]
\[ \text{B2} \]
\[ \text{B3} \]
\[ \text{C1} \]

**Figure 3:** $^1$H NMR spectra of the products obtained from a fatty acid/iron/carbon dioxide/water reaction.
Conclusions

This work aims to develop a better understanding of the TOFA/iron/CO2/water reaction system, which was observed to produce a new type of polymer [28]. Through a combination of different analytical techniques, we have managed to decipher the major reaction pathways of this synthetic process. More work is needed in the future to pin down further details of the reaction. In particular, as we contemplate different applications, variations in the reaction process may be needed to optimize the product properties, and structure-property correlations will be needed. An advantage of this
new ionomer is its low molecular weight that enables it to be readily soluble in a solvent and can be made into different physical forms, such as a solid block, film, or foam.

**Data Availability**

In the manuscript, we clearly stated how and by what instrument and procedure we obtained the data.

**Disclosure**

Mention of trade names or commercial products in this publication is solely for the purpose of providing specific information and does not imply recommendation or endorsement by the U.S. Department of Agriculture. USDA is an equal opportunity provider and employer.

**Conflicts of Interest**

The authors declare that there is no conflict of interest regarding the publication of this paper.

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