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Composition and Structure of Oil Finishing Agent †

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Abstract

FRPs are widely used in the realm of aeronautics, astronautics, aquanautics, geonautics, sports equipment and many more. The subject of the chemical composition of oil finishing agent is seldom found in open literature as it is a proprietary material. Oil finishing agent imparts excellent strength to carbon fibers. Owing to the significant of the composition of the oil finishing agent in the production of carbon fibers, the objective of the current work is to find out in details structure and composition of the oil finishing agent using spectroscopic techniques, such as NMR and FT-IR. The oil finishing agent is known to mainly comprise of three components, namely, the epoxy/amino polyether modified silicone, a non-volatile compound (diluent) and a surfactant. However, the structure of the polyether, nature of the diluent as well as the surfactant were not disclosed. The present study provided insight on such hitherto unknown aspects of the oil finishing agent. Characterization and analysis of the composition of oil agent is complicated and even experienced chemists too are misled owing to the ingenuity with which the oil finishing agent is formulated. NMR (1H, 13C and 29Si) and FT-IR spectroscopy were judiciously used to find that the oil finishing agent is composed of lactic acid (3H, d; 0.81 and 1.21 ppm); formate type anions (1H, s; 8.3 ppm); organo silicone compounds [epoxy polyether modified silicone; hexamethyl disilioxane (HMDS)], a unique and unconventional methanol – dioxane complex used as a non-volatile solvent or diluent (dioxane also serves as surfactant). The presence of four characteristic signals at – 109.12, -22.42, -19.62 and 7.17 ppm in the 29Si NMR spectrum of the oil finishing agent indicated the presence of Si in four different chemical environments in the form of hexamethyl disiloxane (HMDS) and epoxy polyether-modified silicone. Such a result is further supported by the 1H NMR, 13C NMR and FT-IR studies. Thus the mysterious chemical composition of oil finishing agent is disclosed.

Keywords: Oil finishing agent; oil agent; methanol – dioxane complex; diluent; epoxy polyether modified poly organo siloxanes; lactic acid; NMR; FT-IR

INTRODUCTION

It so happened that the research supervisor of the author, namely, Professor Xinling Wang, generously provided a material, to be characterized for the chemical composition, that looked like milk as shown in Figure 1 and the rest is a history.

Figure 1. Image of oil finishing agent that appears to be milk like (Generously provided by Professor Xinling Wang, SJTU, for analysis of the chemical composition of the oil finishing agent)

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The name of the material is oil finishing agent, which is a subject of intellectual property rights and proprietary knowledge. Oil finishing agent is used in the process of production of carbon fibers to impart unusual mechanical strength to the carbon fibers [1]. Usually, the main component of the oil finishing agent is an amino-modified silicone (0.05–10 wt. % amino modification with coefficient of kinematic viscosity of 1000 CST at 25°C) or an epoxy modified silicone (0.05–10 wt. % with a kinematic coefficient of viscosity of 100 CST). In addition, a relatively higher amount (1–90 wt.%) of non-silicone based components are used as diluent, like the higher fatty acid alkyl esters to have the requisite values of the flash point (≥ 70°C) and the coefficient of kinematic viscosity (100 CSI) at 25°C. A copolymer based on acrylonitrile is spun into microfibers and the fibers are dried, compacted and treated with the oil finishing agent. Use of oil finishing agent makes the carbon fibers flame resistant and substantially non-twist. Typically the Si/C ratio in the silicone straight oil finishing agent treated carbon fiber precursors was kept at 0.2 or less [2]. A web of science search (as on 21/12/2024) with the keywords, namely, oil finishing agent, yields, 239 results [3–15]. However, when the keywords, namely, oil finishing agent and carbon fibers, are crossed together, the results condense, by two orders of magnitude, to only 1. It doesn't mean that the oil finishing agent is not exploited well for the production of carbon fibers to impart unusual mechanical strength. But it only means that such a knowledge is not available in the open literature.

Oil finishing agent enables carbon fibers to have excellent strength. The three main components of oil agent include, the epoxy polyether modified silicone, a non-volatile compound and a surfactant. A flow-sheet showing the stage at which oil agent is sued for the treatment of acrylic fibres in the process of carbon fibres production is shown in Scheme 1 [1]. Now the two important questions that remain are: What is the surfactant? What is the non-volatile component used in the oil finishing agent? These questions will be answered in the subsequent sections based on the NMR (^{1}H, ^{13}C and ^{29}Si) and FT-IR analysis of the oil agent. Typical method of production of carbon fibers involving the treatment of the carbon fiber precursor (acrylic fibers) with oil finishing agent comprise of the steps shown in Scheme 1. However much of the knowledge of the function, composition and structure of oil agent is a subject of intellectural property rights analogous to the subject of sizing composition. An attempt has been made to gain new insight into the structure and composition of the oil finishing agent procured from commercial sources that is used for the treatment of carbon fiber precursors prior to the step of carbonization.

EXPERIMENTAL

The oil finishing agent was generously provided by Professor Xinling Wang, SJTU, for the analysis of the chemical composition. NMR (^{1}H, ^{13}C and ^{29}Si) spectra of the oil finishing agent were recorded using a 500 MHz Bruker NMR measuring instrument with $CDCl_3$ as the solvent. FT-IR analysis of the oil finishing agent was carried out in liquid state using a Paragon 1000 FT-IR spectrometer (Perkin Elmer Inc., USA).

RESULTS AND DISCUSSION

Characterization of the Chemical Composition of Oil Finishing Agent Using ^{1}H, ^{13}C and ^{29}Si NMR

The ^{1}H NMR spectrum of oil finishing agent is shown in Figure 2. The term "oil" in "oil finishing agent" tempts one to think that oil finishing agent may appear as a brownish yellow liquid similar to edible or non-edible oils. However, in appearance the oil finishing agent is milky white in colour. When Professor Xinling Wang provided the oil finishing agent to the author for analysis, the first impression is that the sample is analogous to milk. Subsequently, many surprises unfolded as the story goes on. The author is especially grateful to Professor Xinling Wang for entrusting such an astounding area of research filled with many surprises to be found, yet only by those that wish to dwell deeper into the secret things.

A first look at the ^{1}H NMR spectrum by an experienced eye reveals the presence of acetone (3H, s; 2.16 ppm), lactic acid (3H, d; 0.81 and 1.21 ppm) accompanied by the corresponding formate type anion (1H, s; 8.3 ppm) (Figure 2) [16]. Moreover, the signal at 0.08 could be attributed to polydimethylsiloxanes (PDMS) analogous to the poly methyl siloxane compound present in the sizing composition. In addition, a major intense peak at ~ 0 ppm is attributed to the epoxy polyether modified silicone used in the sizing composition [1, 17, 18]. However, in reality the story is not that simple and is more complicated as the subject of composition of oil finishing agent that imparts exceptional strength to carbon fibers leading to revolutionary applications of these materials is often the subject of intellectual property rights. As a result, even an expert spectroscopist will be mistaken and easily be misled by the appearance of peaks and band in the NMR and FT-IR spectra respectively. So a more in-depth insight and unconventional thinking is necessary to gain knowledge on the composition of the oil finishing agent. The questions are as follows: Is the signal at 2.16 ppm indeed due to acetone? If it were to be true there must be an agreement between the ^{1}H NMR and ^{13}C NMR spectra of the oil finishing agent. One would expect to see a peak at 30.9 ppm and 207.07 ppm corresponding to the methyl and carbonyl carbon nuclei of acetone in the ^{13}C NMR spectrum. As expected, the characteristic peak at 30.4 ppm is observed in the ^{13}C NMR spectrum of the oil agent (Figure 3). This makes one very content and lead to commit a mistake in the analysis that the compound present in the oil finishing agent is indeed acetone (note that acetone is a volatile liquid and that the oil finishing agent contains a non-volatile diluent) that is resonating at the chemical shifts of 2.16 ppm in the ^{1}H NMR and at 30.4 ppm in the ^{13}C NMR spectra (Figures 2 and 3).

To verify if indeed, the signals at the chemical shift values of 2.16 ppm in the ^{1}H NMR and at 30.4 ppm in the ^{13}C NMR spectra (Figures 2 and 3) are due to the presence of acetone in the oil finishing agent, the sample of oil finishing agent is subjected to rotoevaporation at 70 °C for 2 h, so as to remove acetone present, if any. Surprisingly, even after attempts for the removal of acetone (if at all present), the signals at the chemical shift values of 2.16 ppm in the ^{1}H NMR (Figure 4) and at 30.4 ppm in the ^{13}C NMR spectra (Figure 5) remained unaltered. This clearly imply that these signals are not due to acetone and but due to some other compound. Then comes the question, what is this unusual compound? The next question follows, what does the signals at 3.62 ppm in ^{1}H NMR and 69.8 ppm in ^{13}C NMR spectra of the oil finishing agent (before rotoevaporation and after rotoevaporation, correspond to (Figures 2–5).

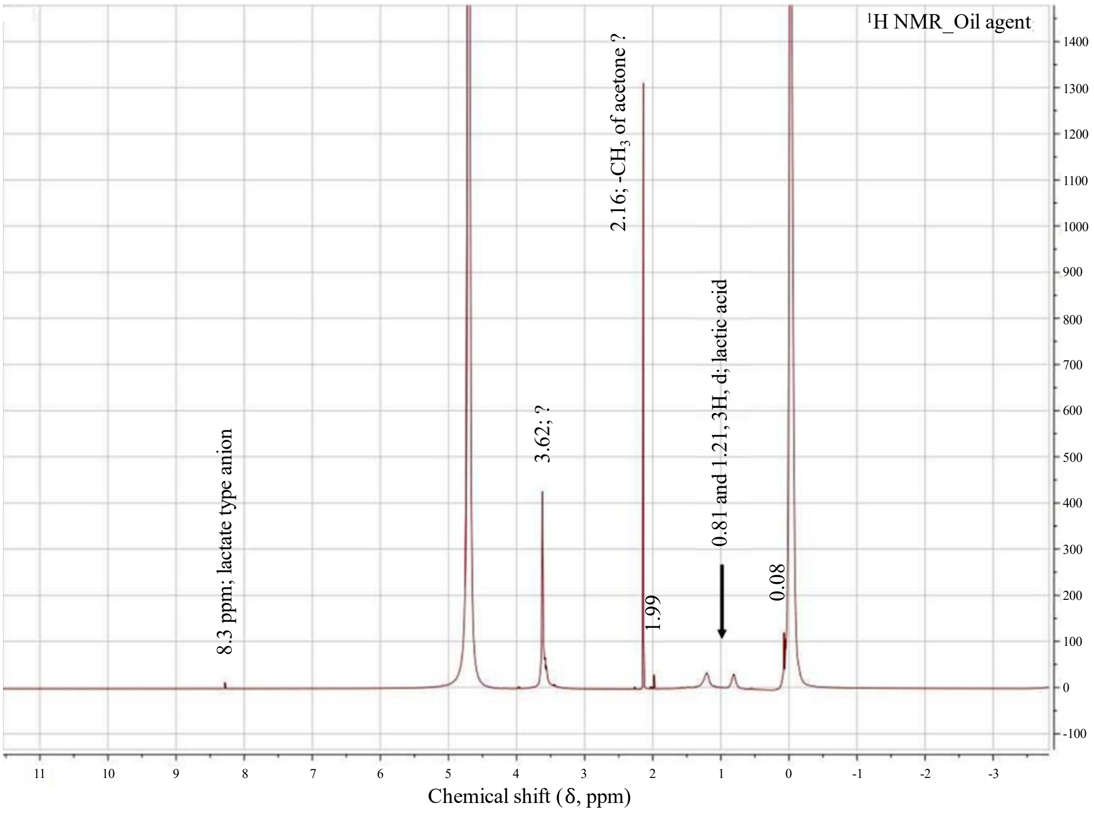

Figure 2. ^{1}H NMR spectrum of oil finishing agent.

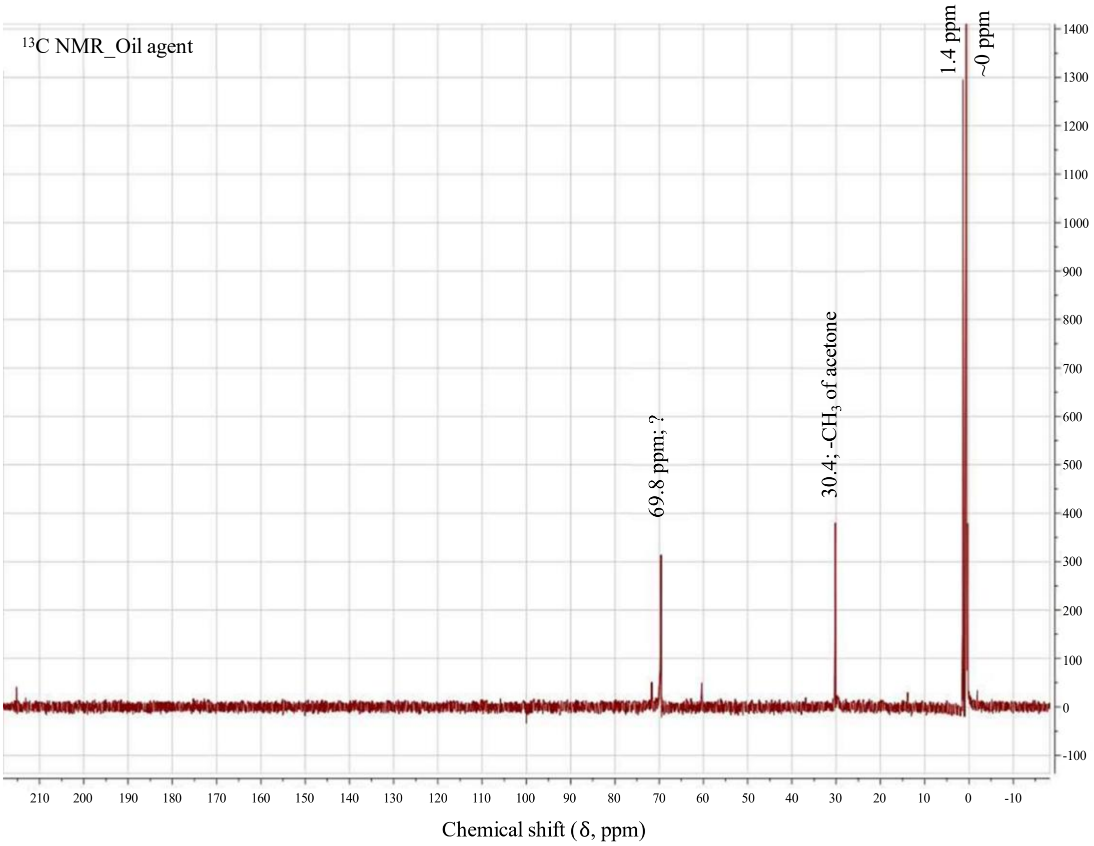

Figure 3. ^{13}C NMR spectrum of oil finishing agent.

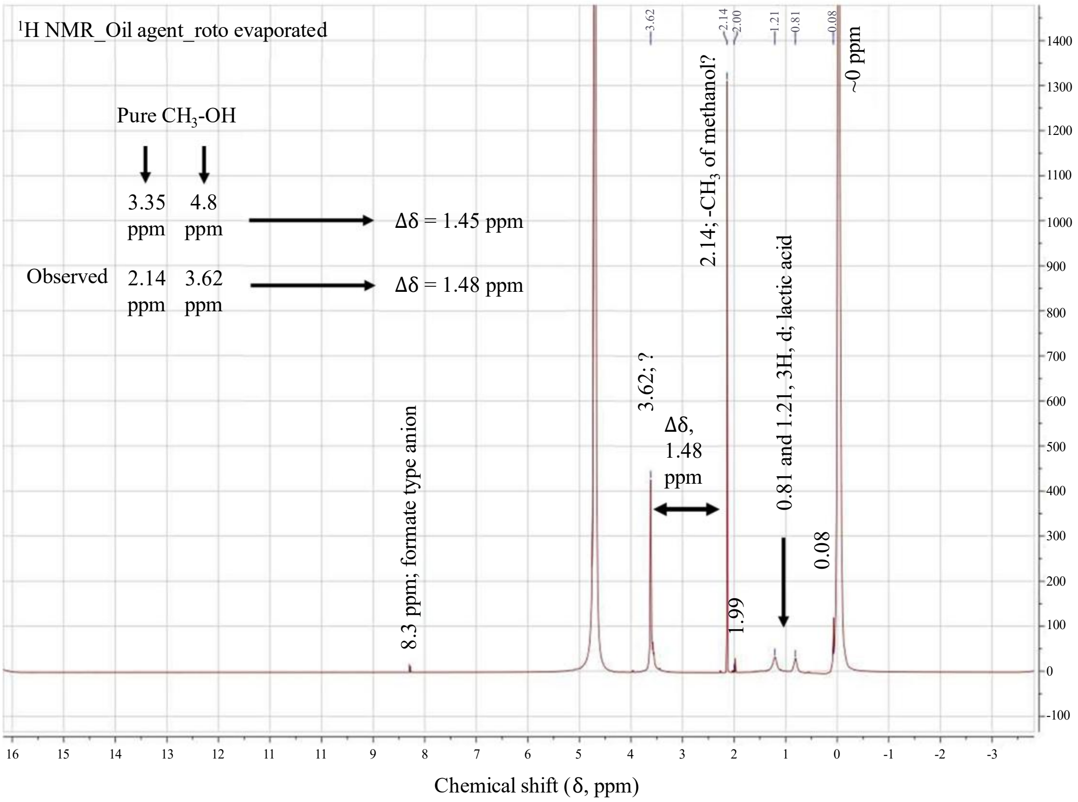

Figure 4. ^{1}H NMR spectrum of oil agent after rotoevaporation at 70°C for 2 h

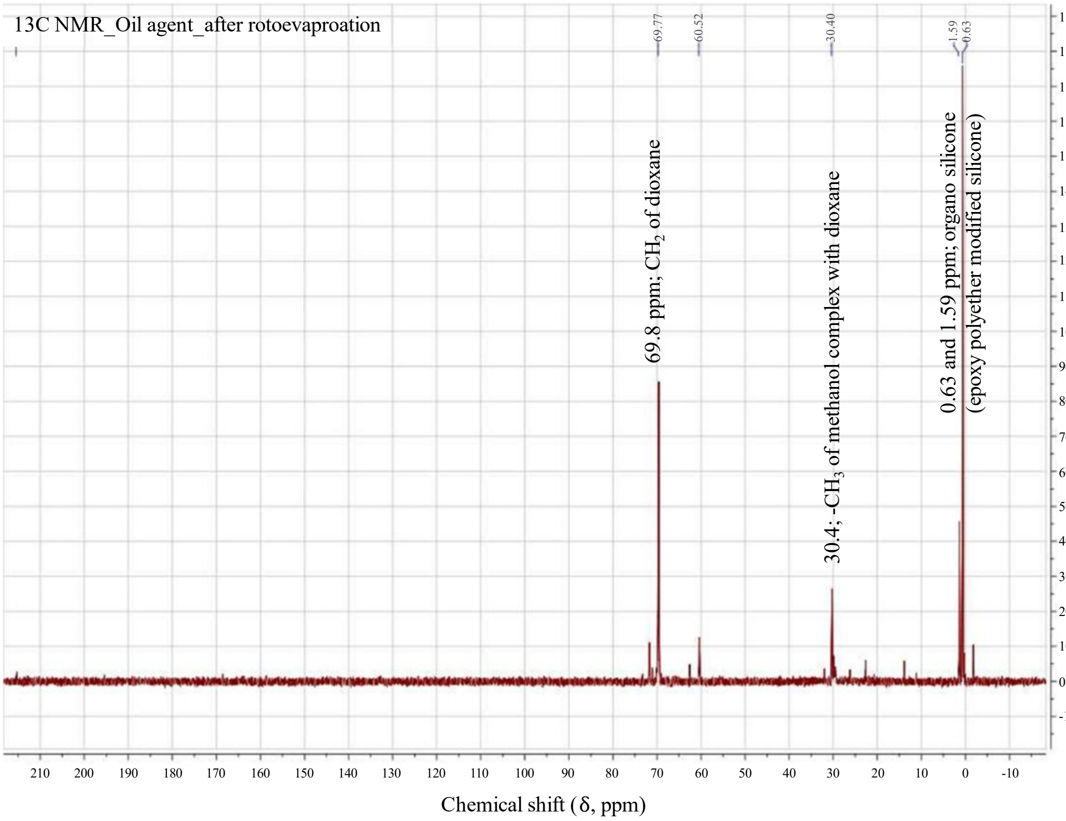

Figure 5. ^{13}C NMR spectrum of oil agent after rotary evaporation at 70 °C for 2 h

Close examination of the ^{1}H NMR spectrum of the oil finishing agent (after rotary evaporation) (Figure 4) reveals that the chemical shift difference between the two signals at 2.14 and 3.62 ppm is 1.48 ppm which is the same as the difference between the signals at 3.35 (3H, S) and 4.8 ppm (1H, S) typical of methanol. Such a chemical shift difference (Δδ, ppm) gives an unmistakable clue that what is present in the oil finishing agent is methanol but in a very peculiar chemical environment. The next valid question would be, if the compound corresponding to the signals at 2.14 and 3.62 ppm (Figure 4) were to be indeed due to methanol, why the peaks do not disappear after rotary evaporation at 70 °C? Another question of vital significance is that what could have caused the shift in the signals of methanol to such a large extent (Δδ, 1.48 ppm)? There could be extensive hydrogen bonding between methanol and some novel oxygen containing solvent used in the oil finishing agent that might have led to an unusual enhancement of the boiling point of methanol and also could have simultaneously caused an unusual up field shift in the signals of methanol. It is known that hydrogen bonding interactions between methanol and water to contribute to such up field shift and elevation of boiling point of methanol from 64.7 to as high at 95 °C. This raises the next question on the nature of the peculiar solvent used in the oil finishing agent. From the knowledge gained on the structure and composition of sizing agent using NMR and FT-IR spectroscopic studies [17, 18], it is found that the crown ethers, that are generally used as dispersing agents for carbon fibres, resonate at the chemical shift value of 3.6 ppm. This gives a clue that the peak in question at 3.62 ppm is not just due to a single compound, namely, methanol, but due to a combination of two compounds, namely, alcoholic protons of methanol as well as the methylene protons of crown ethers. The next question would be, if the crown ether used in the composition of oil finishing agent is the same as that of the crown ether used in sizing composition (namely, 18 crown 6 ether, as deduced from the NMR analysis) [17, 18]. However, Dr Dandan Zhu, the research co-worker and guide of the author raised serious concerns and has watered down all these unconventional arguments, citing the only good reason that the relative integral area under the peaks at 2.14 and 3.62 ppm (Figure 6) is not 1:0.33 as expected in the case of methanol but was 1:3. Thus, based on the reliable observations from the NMR analysis, it is surmised that indeed the oil finishing agent contained a peculiar complex of methanol and an unusual oxygen containing solvent like crown ether (dioxane) that has caused the up field shift of the signals of methanol. Moreover, if a crown-ether as simple as dioxane were to be present in the composition of the oil finishing agent, the ratio of integral areas of the signals at 2.14 and 3.62 ppm will for sure be 1:3 and not 1:0.33. In fact, as shown in the ^{1}H NMR spectrum of the oil finishing agent after rotary evaporation, the ratio of the integral areas of the signals at 2.14 and 3.62 ppm is 1:311 (Figure 6). This proves beyond doubt that the peak in question at 3.62 ppm is not just due to the alcoholic protons of methanol (1H, s; 3.62 ppm) but also due to the 8 methylene protons of the dioxane (8H, S; 3.62 ppm). Formation of extensive hydrogen bonding interactions and a one to one complex between methanol and dioxane are well documented in literature [19]. Moreover, a peak at 69.8 ppm in the ^{13}C NMR spectrum of the oil finishing agent after rotary evaporation also point to the presence of dioxane used as non-polar solvent in the oil finishing agent composition. Dioxane is a well-known surfactant [20] and patent literature clearly gives the clue that the composition of the oil finishing agent comprises of surfactant as well, in addition to a non-volatile solvent (in the present case a complex of methanol and dioxane) [1]. So dioxane could have been serving the purpose of surfactant as well, in addition to being used as a non-polar solvent forming complex with methanol resulting in a non-volatile solvent (diluent). However, the author leaves to the discretion of the expert scientists in the field to validate the truthfulness of the arguments. The arguments regarding the presence of lactic acid in the composition of oil agent based on the signal (3H, d; 0.81 and 1.21 ppm) in the ^{1}H NMR spectrum of oil finishing agent (before rotary-evaporation and after rotary evaporation) (Figures 2 and 4) are as follows: though the peaks corresponding to lactic acid at 20 , 66 and 187 ppm attributed to methyl, methine and carboxylic groups, are expected in the ^{13}C NMR spectrum of the oil finishing agent (before rotary evaporation and after rotary evaporation) (Figure 3 and 5), none of these signals are observed. That does not mean that the lactic acid, as evidenced by the ^{1}H NMR spectra (3H d at 1.21 ppm) (Figures 2 and 4) is absent. In contrast, since the signal intensity corresponding to lactic acid is very low in ^{1}H NMR spectrum

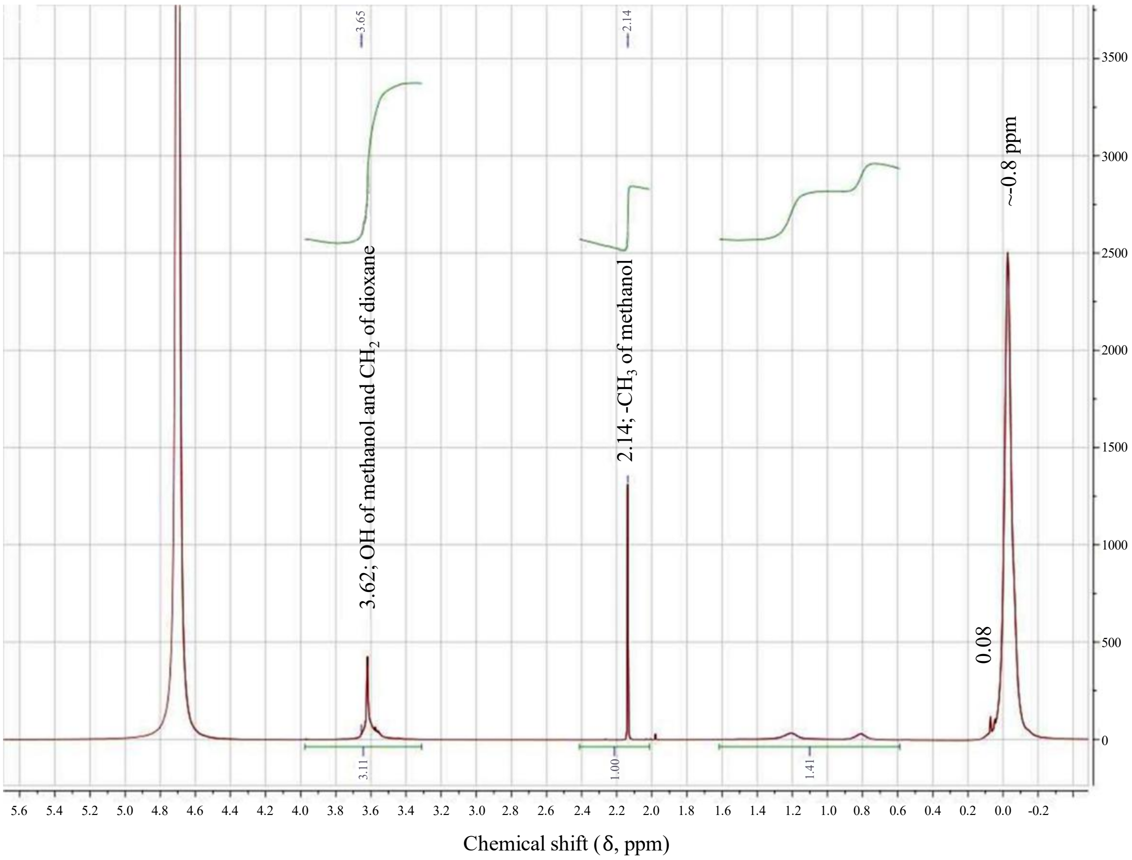

Figure 6. ^{1}H NMR spectrum of oil finishing agent after rotary evaporation at 70°C for 2 h (with integral areas of the signals at 2.14 and 3.62 ppm marked)

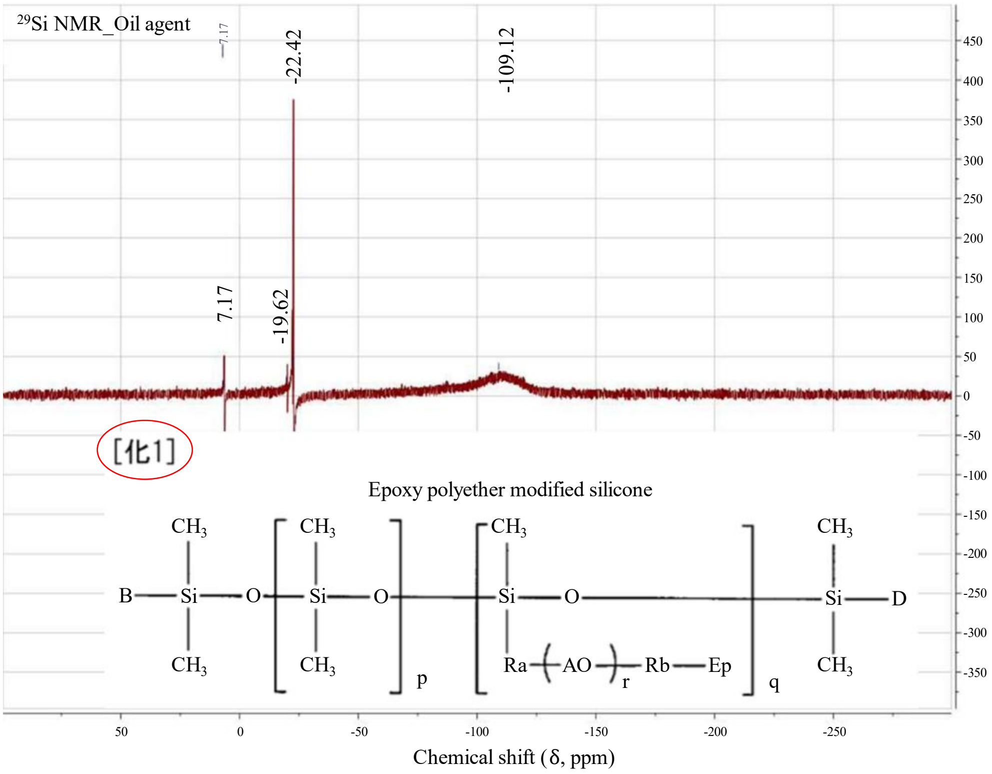

Figure 7. ^{29}Si NMR spectrum of oil finishing agent

itself, lactic acid will obviously be almost invisible in ^{13}C NMR spectra of the oil finishing agent (Figure 3 and 5) as ^{13}C NMR spectroscopy is nearly 5000 times less sensitive than ^{1}H NMR spectroscopy. This should not in any way mean that ^{13}C NMR is unreliable as some expert emeritus professors mistake. ^{13}C NMR is as reliable as ^{1}H NMR except that the sensitivity of ^{13}C NMR is four orders of magnitude lower than that of the ^{1}H NMR spectroscopy.

The intense signals at 0.08 and -0.08 ppm in the ^{1}H NMR spectra of the oil finishing agent before and after rotary evaporation (Figures 2 and 4) are attributed to the presence of hexamethyl disiloxane (HMDS) [21] and epoxy polyether-modified silicone [1] respectively. Corresponding peaks in the ^{13}C NMR spectra of the oil finishing agent before and after rotary evaporation were observed at 1.4 and ~0 ppm (Figures 3 and 5). The presence of organosilicone compounds like HMDS and epoxy ether modified silicone are evident from the ^{29}Si NMR spectrum of oil finishing agent where in the presence of Si in four different chemical environments results in four signals at the chemical shift values of -109.12, -22.42, -19.62 and 7.17 ppm (Figure 7).

Characterization of the Chemical Composition of Oil Finishing Agent Using FT-IR Spectroscopy

Analogous to NMR spectra (Figures 2–7) of the oil finishing agent, the FT-IR spectrum of oil finishing agent too hold vital information on the composition of the oil finishing agent. As revealed from the NMR analysis of the oil finishing agent, the composition of oil finishing agent contained epoxy polyether modified silicone showing major and intense signal close to ~ 0 ppm corresponding to Si-CH_3 bonds. Likewise, the FT-IR spectrum of oil finishing agent too reflected the presence of polysiloxanes in the composition of the oil finishing agent. Four characteristic bands at 798 cm^{-1}, 1023–1095 cm^{-1} (doublet), 1260 cm^{-1} and 2963 cm^{-1} were noticed. The sharp intense band at 798 cm^{-1} corresponds to the stretching vibration of -Si-O- bond in the organosilicone (polysiloxane) compound in the oil finishing agent. The doublet in the range of 1023–1095 cm^{-1} is attributed to the Si-O-Si stretching vibrations in groups like R_3-Si-O-Si-R_3. The band at 1260 cm^{-1} is attributed to the stretching vibration of –Si-CH_3- bonding network. The band at 2963 cm^{-1} is attributed to the C-H stretching of CH_3 group bound to Si (Figure 8) [22, 23].

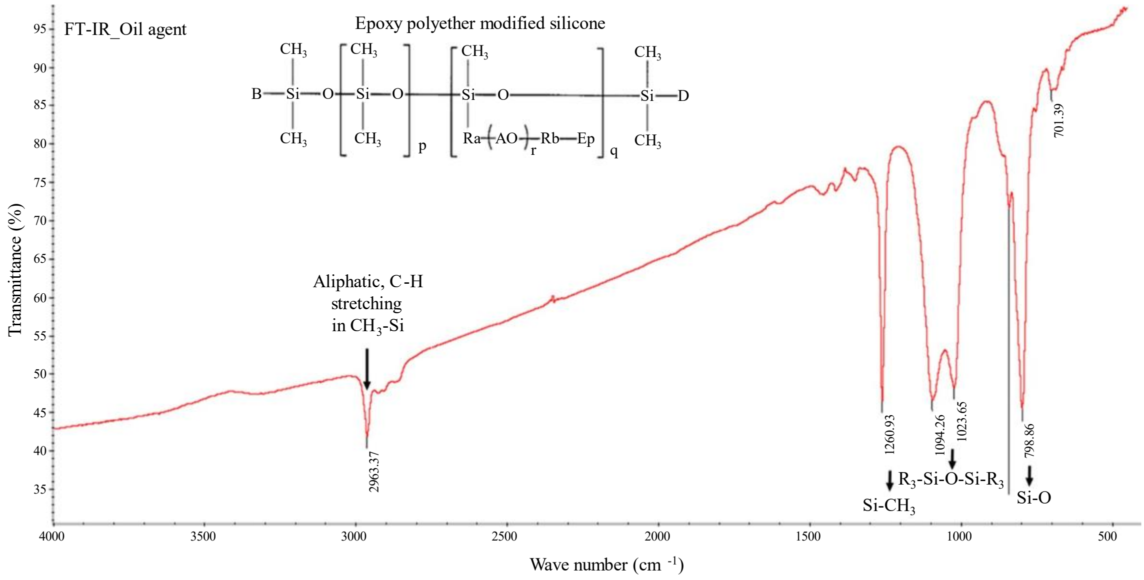

Figure 8. FT-IR spectrum of oil finishing agent

Thus the oil composition contained the inevitable presence of organosiloxane dispersed in the methanol-dioxane complex solvent. Unlike the sizing composition, the oil composition is non-aromatic as no signals characteristic of benzene ring are observed in the region of 1500–1620 cm^{-1}. The FT-IR spectra features are indeed mysterious that the spectrum is simple rather than complex as expected. Even though, the spectral bands typical of methanol, 1, 4-dioxane and epoxy ring are expected to be seen they were not observed and only features typical of polysiloxane are visible (Figure 8). The finished product, namely, carbon fibers, (both pitch based and PAN based), produced using oil finishing agent with unusual mechanical strength were thoroughly characterized using XRD, Raman and XRM for gaining insight into their structure. The readers are advised to consult the same for further understanding the impact of treatment with oil finishing agent on the structure and properties of the carbon fiber materials, apart from details on the process of sizing that induces polarity on the carbon fiber surface [24–27].

CONCLUSION

Use of oil finishing agent for the treatment of carbon fiber precursors in the production process of CFs imparts unusual mechanical strength to the carbon fibers in general, and to the composites formed there of via the formation of strong interface with R-Si-O-Si-R type linkages at the interface. NMR (^{1}H, ^{13}C and ^{29}Si) spectroscopic studies on the structure and chemical composition of the oil finishing agent revealed the presence of methanol-dioxane complex as the unusual solvent (diluent) used in the oil finishing agent to disperse the epoxy modified organo polysiloxanes. Traces of lactic acid were also observed in the oil agent composition. ^{1}H NMR spectra of the oil agent revealed methanol-dioxane complex as the major component of the oil finishing agent, only next to the epoxy polyether modified silicone. Polyorgano siloxane type compounds, as those used in the sizing composition, namely, polydimethyl siloxane (PDMS) and hexamethyl disiloxane (HMDS) were also present in the oil finishing agent. Such organ siloxane compounds as well as the epoxy polyether modified silicone compounds on the surface of the CFs are vital for the formation of the strong chemical bonding network between the CFs and the filling matrix (resins) facilitating the formation of strong interface. FT-IR spectrum of the oil finishing agent proved the presence of epoxy polyether modified silicone with Si-O, Si-CH_3 and R-Si-O-Si-R type of bonding network. Thus, NMR and FT-IR spectroscopic techniques are judiciously and unmistakably used to unravel the mysterious chemical composition of the oil finishing agent which is a subject of commercial significance. Aspects such as the mechanism of action of oil finishing agent in imparting unusual mechanical strength to the carbon fibers as well as the unique chemical species imparting such unusual mechanical strength are yet to be found.

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