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Fourier Transform Infrared Characterization of Construction Joint Sealants

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ABSTRACT

This paper presents the results of the FTIR (Fourier-Transform Infrared) spectroscopy study of commercially available silicone-based sealant materials commonly used for sealing voids, cracks, and joints in non-structural building components of commercial and residential buildings. The sealants prevent moisture leakage through the parts, making the building envelope air and water-tight, sustainable, and energy-efficient. FTIR spectroscopy is a widely used technique in characterizing pharmaceutical products, mineralogical compositions, forensic analysis, food, chemical, semiconductor, petroleum, and agro-industries. This work demonstrates the application of FTIR analysis in the study of construction materials. It reports the IR (Infrared) spectral signatures of the selected building joint sealants used in the construction industry. It is found that several IR bands are common to most of the samples, suggesting that there is a reasonable similarity in the molecular composition of the three different manufacturer brands, including DAP (Dicks-Armstrong-Pontius), GE (General Electric), and HDX (Home Depot Product). Despite the multiple similarities in the IR bands of the samples studied, the FTIR techniques provided enough evidence to distinguish the samples and suggest that the composition and molecular structures of most of the silicone brands possess unique IR signatures.

Keywords: FTIR analysis of building materials, Silicone joint sealant, Characterization of construction materials, FTIR bands of building sealants, Characterization of silicone sealants, FTIR spectroscopy.

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1. INTRODUCTION

Silicone is produced in the form of elastomers, fluids, gels, and fluids. These products have been used for various purposes in different sectors, including construction, automotive, electrical and electronics, pharmaceutical, and other industries [1-4]. The hydrophobic nature of silicone rubber also makes it one of the primary materials of choice for coating materials and parts exposed to varying environmental conditions [4]. In 2018, the construction industry was the major consumer of silicone, followed by transportation (aerospace and automotive) and electrical and electronics industries. In the coming years, the market share of silicone in the construction sector is projected

to grow globally [5]. Despite its gradual aging and degradation, in building construction, silicone is a widely used hydrophobic polymer having excellent weathering resistance and durability [1]. The applications include sealing voids, cracks, and joints in non-structural building components (windows and door frames, between panels), which prevents moisture leakage through the parts, making the building envelope air and water-tight, sustainable and energy-efficient construction [1,2]. Sealed joints and cracks in building envelope also minimize smoke leakage to adjacent rooms and buildings [6], retain heat within the building envelope, and reduce energy use and utility cost [1].

Silicone sealants contain thermally stable rubber called polydimethylsiloxane (PDMS), which possesses very stable chemical bonds of –Si-O-, -Si-C- and –Si-H [7]. Crosslinking, which is a formation of a molecular bond between silicone sealant polymer chains, starts when the sealant is squeezed out and interacts with the atmospheric moisture and the moisture on the joint or the building's surface components [1]. These materials' benefits include their reasonable resistance to UV, chemical and high-temperature degradation, color stability, and flexibility. The unpleasant odor and poor adhesion of paints on the silicone surfaces are some of the shortcomings [7]. Research works demonstrated the wide range of FTIR spectroscopy applications for the characterization of construction materials [7-15]. Some of the benefits of using this technique include the reliability of the analytical results collected in a short time with a relatively inexpensive system, and the simple sample preparation required [7-12]. A combination of infrared spectroscopy, x-ray diffraction, and scanning electron microscopy was also used to identify mineralogical compositions of construction materials used for plastering and foundations of ancient buildings [8]. The use of IR spectroscopy in the study, and spectral assignment of cementitious materials including ordinary Portland cement, calcium aluminate cement primarily used to resist sulfate attack, and calcium sulfoaluminate cement used where fast setting and high early strength of concrete is desired, is presented elsewhere [9]. FTIR spectroscopy is a valuable tool to detect the presence of asbestos and quantify the amount present in building materials with a detection limit of 1% w/w [10,11]. Its application as a potential technique for mapping and evaluating fire damage in buildings and as a non-destructive tool for assessing the extent of thermal damage and concrete degradation was also demonstrated [12]. One of the various applications of silicone sealants as a construction material in civil infrastructure is as an expansion joint in railway

construction [16]. Li et al. (2018) used FTIR to study ultraviolet light's effect on the degradation of silicone sealant used for railway-track expansion joints. To study the effect of Al₂O₃ fibers on silicone's thermal stability for aerospace application, Yao et al. (2014) used FTIR to verify the hydroxyl content of the Al₂O₃ fiber loadings [17]. The FT-IR investigation results indicate that hydroxyl content on Al₂O₃ surface is reduced at 900 °C compared with the samples dehydrated at 500 °C and as-received. FTIR has also been used as a valuable non-destructive tool to evaluate the extent of thermal damage and degradation of concrete, with a potential application for forensic analysis of building's fire damage [12]. In this study, the spectral signatures of three different brands of a dozen commercially available silicone building sealants are characterized using Fourier-Transform Infrared Spectroscopy.

The use of the FTIR technique in the study of construction materials has been primarily focused on the study of Portland cement [12-15] and asbestos materials [10,11]. With the wide range of benefits, the FTIR technique to offer to the construction materials research and industry, the application of this technique in the study and analysis of other construction materials is limited. This study demonstrates the FTIR system's suitability and sensitivity for analyzing materials used in the construction industry. We believe that the present work will inspire the use of FTIR spectroscopic technique for qualitative analysis of building materials including in quality control, verifying the materials' compliance with the required specification on-site, identification of counterfeit products, and evaluation of the gradual degradation of construction materials due to aging and environmental factors. To this end, the work presented in this article corroborates the significance of this technically sound approach for construction materials research and contribute to the body of knowledge in the study of building materials.

2. MATERIALS AND METHODS

Table 1 summarizes the details of the three brands of the commercially available silicone sealants commonly used for sealing voids, cracks, and joints for indoor and outdoor surfaces in commercial and residential buildings, making the building envelope air and water-tight. The sealants fill the spaces between non-structural building components such as windows and doorframes, between panels, sidings, kitchen, bath, and plumbing fixtures. Per the manufacturer's description of the sealants' principal ingredients, sample S1 contains titanium dioxide, ethylene glycol, and calcium carbonate and meets latex sealant ASTM C-834, standard specification [18]. Sample S2, with the same manufacturer as S1, contains titanium dioxide, propylene glycol, acrylic polymer mixture, calcium carbonate, diisononyl phthalate, and other additives, and meets the ASTM C-920 standard specification for elastomeric joint sealants [19]. Sample S3 contains titanium dioxide, quartz, 50-70 percent by weight of limestone, diethylene glycol dibenzoate, and petroleum distillates [20]. Similarly, sample S4 contains titanium dioxide, quartz, 50-70 percent by weight of limestone,

Diethylene glycol dibenzoate, petroleum distillates, and carbon [21]. A typical picture showing the various silicone-based joint sealant applications in construction is provided elsewhere [22]. FTIR spectra of the silicone samples were acquired using Nicolet™ iS™ 10 FTIR Spectrometer (Thermo Scientific Inc., Madison, WI, U.S.A.) at room temperature in the frequency range of 4,000 to 400 cm⁻¹ using 32 scans at a 4 cm⁻¹ resolution. The FTIR system is equipped with KBr/Ge coated beam splitter and Deuterated Triglycine Sulfate detector and OMNICTM standard spectroscopy software to collect and process the spectral data. The twelve different types of joint sealants identified for the study are representative of the three manufacturer brands widely used in the residential and commercial building construction industries. The 2 x 2 x 1 cm size specimen of the products described in Table 1 was prepared and allowed to be cured at room temperature. The dried samples were loaded to ensure adequate contact between the specimen and the crystal. The incident beam interacts with the specimen molecules, and the frequency of the beam matching with the

molecular vibration is absorbed. The intensity of the IR bands is dependent on the rate of change of the dipole moment, with the larger the change in the dipole moment of the bond

resulting in a considerable vibration of the bonds and larger IR band intensity [23].

Table 1. Description of the samples.

| *Description of the Samples | Sample # | Brand Name | ASTM Standard |
|---|----------|------------|--------------------|
| Latex plus silicone caulk | S1 | HDX | Meets ASTM C-834 |
| Premium elastomeric plus silicone caulk | S2 | | Meets ASTM C-920 |
| Alex Fast dry acrylic latex caulk plus silicone | S3 | *DAP | Exceeds ASTM C-834 |
| Alex plus acrylic latex caulk plus silicone | S4 | | Exceeds ASTM C-834 |
| Silicone max, 100% silicone | S5 | | Meets ASTM C920 |
| Kiwi seal ultra-premium siliconized sealant | S6 | | |
| Kiwik seal plus | S7 | | |
| UV resistant, 100% silicone sealant, | S8 | GE | |
| Silicone 2+ , 100 % silicone | S9 | | |
| Silicone 1+ , 100 % silicone | S10 | | Meets ASTM C-920 |
| GE paintable and waterproof | S11 | | Meets ASTM C-920 |
| GE Paintable quick dry | S12 | | Exceeds ASTM C-834 |

3. RESULTS AND DISCUSSION

The FTIR spectra of the three brands of the silicone sealant studies are shown in Figure 1, and the complete IR-bands of silicone-based building sealants are summarized in Table 2. In general, the IR bands located at about 890-860 cm-1 and 1100-1000 cm-1 indicate the presence of Si-H bending [24] and Si-O-Si stretching modes [24], respectively. The IR spectra of the HDX brands of the sealants labeled as samples S1 and S2 shown in Figure 1(a) have characteristic prominent IR bands located at 1727 cm-1, 1154 cm-1, 871 cm-1, 711 cm-1. The three IR bands which exist in sample S1 but not in sample S2 are the bands located at 1394 cm-1, 2024 cm-1, and 2962 cm-1. Samples S1 and S2 possess a strong IR band at 711 cm-1 region, indicating the presence of CH3 asymmetric rocking and Si-C asymmetric modes, and the IR band at 2163 cm-1 of the sample S1 suggests the presence of CH3 asymmetric stretching mode, and the IR band at 1024 cm-1 in sample S2 indicates the presence of Si-O-Si symmetric stretching in the sealant sample [25]. As described in Section 2 above, the ingredients of samples S2 are slightly different from sample S1, which resulted in more IR bands in sample S2 than in sample S1, including 745 cm-1, 962 cm-1, 1111 cm-1, 1241 cm-1, 1341 cm-1, 1379 cm-1, 1417 cm-1, and 2874 cm-1.

Figure 1 (b) shows the FTIR spectra of five different samples (S3-S7) of the DAP brand silicone-based construction sealants studied. As shown in Table 2, The IR bands located at 2922 cm-1, 1727 cm-1, 1154 cm-1, 871 cm-1, and 711 cm-1 are common to samples S3, S4, and S7. Again the 711 cm-1 band in samples S1, S2, S3, S4, and S7 confirms the presence of CH3 asymmetric rocking and Si-C asymmetric modes. Samples S5 and S6 appeared to have different characteristic IR bands among each other and the other samples within the same brands. The three prominent IR bands of sample S5 are

located around 1258 cm-1, 1008 cm-1, and 785 cm-1, and the other three weak Ir bands are at 2962 cm-1, 700 cm-1, and 472 cm-1. The IR bands at 700 cm-1 and 2962 cm-1 regions indicate the presence of Si-C symmetric stretching and CH3 asymmetric stretching mode in sample S5 of the silicone sealant [25]. As shown in Table 2, sample S6 has several IR bands with a peak at 1723 cm-1 being very strong and sharp. Figure 1(c) shows the IR spectra of five samples (S8-S12) selected from silicone-based GE brand construction joint sealants. As can be seen, samples S8, S9, and S10 appeared to have similar IR bands located at 2963 cm-1, 1258 cm-1, 1008 cm-1, and 785 cm-1. The weak IR band at 2963 cm-1 indicates the presence of CH3 asymmetric stretching mode in these samples. On the other hand, samples S11 and S12 possess characteristic IR bands located around 1411 cm-1, 1099 cm-1, 872 cm-1, and 711 cm-1. The bands at 1411 cm-1, 1099 cm-1, and 711 cm-1 indicate the presence of CH3 asymmetric bending, Si-O-Si asymmetric stretching, and Si-C asymmetric modes, respectively. The IR bands located at 863 cm-1 and 685 cm-1 are also common to samples S9 and S10, indicating the presence of CH3 symmetric rocking mode in these samples. The IR band located at 2963 cm-1 exists in Samples S1, S5, S6, S8, S9, and S10, indicating the presence of CH3 asymmetric stretching mode in these samples. The IR band located at 1728 cm-1 is common to some of the samples across the three different brands of the joint sealants, including samples S1, S2, S3, S4, S7, and S12. Similarly, the bands located at 711 cm-1, 872 cm-1, and 1024 cm-1 also exist in some of the samples across the construction sealant's three brands. The appearance of these characteristic IR bands in HDX, DAP, and GE brand silicone-based construction sealants indicates common ingredients in the joint sealants' production across the three manufacturers considered.

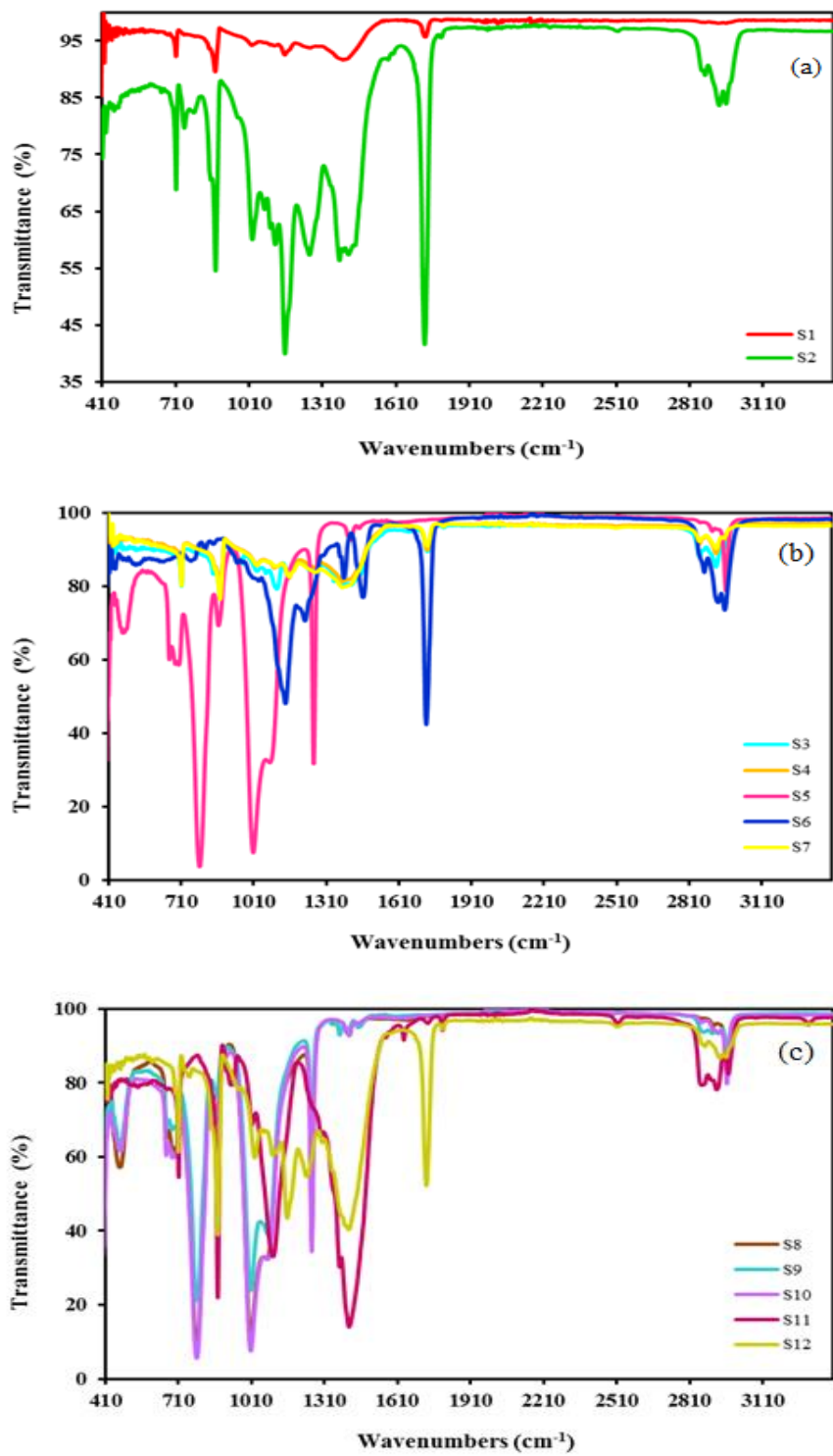


Figure 1. FTIR spectra of HDX (a), DAP (b), and GE (c) brands of silicone sealants used in residential and commercial buildings.

Table 2. IR-bands of silicone-based building sealants.

| IR Absorption Bands, cm ⁻¹ | | | | | | | | | | | |
|---------------------------------------|------|------|------|------|------|------|------|------|------|------|------|
| S1 | S2 | S3 | S4 | S5 | S6 | S7 | S8 | S9 | S10 | S11 | S12 |
| 711 | 711 | 711 | 599 | 472 | 1141 | 711 | 468 | 468 | 472 | 711 | 711 |
| 872 | 745 | 872 | 711 | 700 | 1222 | 872 | 700 | 685 | 685 | 872 | 872 |
| 1021 | 873 | 962 | 872 | 785 | 1382 | 1022 | 785 | 785 | 787 | 927 | 1024 |
| 1156 | 962 | 1024 | 1023 | 1008 | 1462 | 1156 | 1008 | 863 | 863 | 1099 | 1097 |
| 1394 | 1024 | 1106 | 1095 | 1258 | 1723 | 1263 | 1258 | 1008 | 1008 | 1372 | 1157 |
| 1730 | 1111 | 1155 | 1155 | 2962 | 2873 | 1376 | 2962 | 1079 | 1258 | 1412 | 1237 |
| 2024 | 1155 | 1273 | 1267 | | 2928 | 1728 | | 1258 | 2962 | 2921 | 1410 |
| 2163 | 1241 | 1341 | 1377 | | 2957 | 2852 | | 2963 | | | 1729 |
| 2960 | 1341 | 1407 | 1728 | | | 2922 | | | | | 2933 |
| | 1379 | 1728 | 2853 | | | | | | | | |
| | 1417 | 2923 | 2922 | | | | | | | | |
| | 1727 | | | | | | | | | | |
| | 2874 | | | | | | | | | | |

4. CONCLUSION

The study used FTIR spectroscopy to characterize silicone-based construction joint sealants used in commercial and residential buildings. The samples studied include a dozen commercially available HDX, DAP, and GE brands silicone-based sealants. As shown in [Figure 1](#) and [Table 2](#), most of the samples have characteristic IR bands suggesting some level of similarity in composition and molecular structures of the joint sealants produced by different companies. However, a detailed analysis of the IR spectral signatures of each sample indicates that there is a clear distinction between the samples, and none of the samples is identical to each other. The results shown in [Figure 1](#) reveal a wide range of IR band intensity variations ranging from a very broad and weak to very strong and sharp IR peaks. The observed band intensities are proportional to the amount of the constituent material available [\[24\]](#) in the sample. In general, the IR bands located at about 890-860 cm⁻¹ indicates the presence of Si-H bending

vibrational mode in the sample [\[24\]](#). Accordingly, except in the DAP brand samples S5, S6, and S8, and GE brand sample S11, Si-H bending mode appeared in the remaining eight samples. Similarly, the IR bands located at about 1100-1000 cm⁻¹ indicates Si-O-Si stretching mode [\[23\]](#), which appeared in all samples except S6. The IR band located at about 871 cm⁻¹ is observed to be the strongest and very sharp across most of the samples, including S1, S3, S7, S11, and S12. For samples S2 and S12, there is a strong and sharp peak at around 1727 cm⁻¹. For samples S5, S8, S9, and S10, the prominent IR band is located at around 785cm⁻¹.

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AUTHORS CONTRIBUTION

This work was carried out in collaboration among all authors.

CONFLICT OF INTEREST

The author declared no potential conflict of interest or conflict of interest.

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