Bulletin of Electrical Engineering and Informatics
Vol. 13, No. 2, April 2024, pp. 1433~1442
ISSN: 2302-9285, DOI: 10.11591/eei.v13i2.6070

Effects of processing parameters on the leakage current of silicone rubber insulator

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Article Info

Article history:

Received Feb 23, 2023
Revised Nov 27, 2023
Accepted Dec 20, 2023

Keywords:

Fourier transform infrared
High voltage insulator
Leakage current
Processing parameters
Silicone rubber

ABSTRACT

Silicone rubber (SiR) is known for its exceptional electrical insulation properties. The performance of SiR could be affected by many factors, including processing parameters, particularly mixing speed and time. While these parameters are crucial for ensuring the homogeneity of blended polymeric materials, their electrical impact remains relatively unexplored. This research investigates the effect of varying processing parameters on SiR samples during rapid aging under the incline plane tracking (IPT) test. The study unfolds in three phases, with the final IPT stage revealing the significant influence of different mixing speeds and times on the recorded leakage current (LC) values for each sample. Sample 2, subjected to 70 rpm mixing speed and 10 minutes of mixing time, exhibited great resistance to tracking and erosion. Fourier transform infrared spectroscopy (FTIR) was conducted on the samples before and after the IPT test to further analyze the impact of the varying processing parameters. Once again, sample 2 displayed notable resilience, demonstrating lower reductions in absorbance values for key functional groups. In conclusion, the specific processing parameters of 70 rpm and 10 minutes have been shown to positively influence the performance of SiR, enhancing their resistance to tracking and erosion during rapid aging.

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1. INTRODUCTION

Polymers like silicone rubber (SiR) have gained prominence in the outdoor high voltage (HV) insulation industry owing to their exceptional potential. SiR possesses excellent physical and dielectric properties [1]. These characteristics allow SiR to have high resistance against moisture and contaminants. However, continuous exposure to environmental stresses, which include humidity and pollutants, exposed these polymeric outdoor insulators to dry band arching (DBA), leading to tracking and erosion. Over time, these occurrences will lead to the aging and degradation of SiR [2]. Hence, considerable research has focused on enhancing the resilience and performance of polymeric materials, including the incorporation of fillers to strengthen polymeric insulators. The effectiveness of fillers varies based on their type, size, distribution, surface area, and chemistry. While certain fillers primarily increase the polymer volume, others establish chemical bonds with the polymer matrix, leading to interactive effects [3]. Alumina trihydrate (ATH) filler is

commonly preferred to suppress tracking and erosion in SiR. ATH effectively slows the decomposition rate by releasing water, which dilutes combustible gas, slows temperature rise, and prevents ignition [4]. This helps to suppress DBA and corona, hence the tracking and erosion of polymers. Few concentration ranges have been highlighted as optimum values for micro-sized ATH, ranging from 40 wt% up to 60 wt% [5]–[7].

Essentially, the inclusion of fillers in HV outdoor polymeric insulators enhanced specific properties and led to cost reductions [8]. However, current research focuses on nano-sized fillers, which hold promising potential but are not cost-effective. Cherney [9] mentioned that using nano-sized filler in outdoor insulation applications had been limited due to its expensive cost. Moreover, the intricate processing method, easy agglomeration, and higher cost were the reasons for the recent comeback of micro-sized filler. Recent studies [10]–[12] have started to combine micro and nano-sized fillers. It is essential to recognize the continued relevance and cost-effectiveness of micro fillers, which have a proven track record and require less extensive studies. Instead of changing the filler, exploring the improvement of SiR by focusing on its processing parameters with micro filler could be the next best alternative to using nano fillers or combining both fillers. The knowledge gaps addressed in this study were identified by examining the key findings related to the impact of processing parameters in the existing literature and their significance in the context of HV insulation. Table 1 summarises the literature concerning processing parameters and their contributions over the years, regardless of the base material.

Table 1. Summary of previous findings related to processing parameters

Ref	Year	Key findings
[13]	2013	The samples of SiR containing nano and micro silica were prepared using conventional mechanical mixing and electrospinning. The comparison between the two processing parameters showed that electrospun samples had greater erosion resistance.
[14]	2016	The processing parameters (mixing speed and time) and filler concentration significantly influenced the surface resistivity value of the SiR insulator.
[15]	2017	It was highlighted that optimizing the formulation of the material by adjusting the processing parameters (mixing speed and time) and filler concentration could significantly influence the surface resistivity and relative permittivity of the produced SiR insulating material.
[16]	2018	In rubber processing, increasing filler loading or the rotor speed reduces the impact of mixing time on the dispersion index and the Payne effect. This indirectly affects the rubber's conductivity by influencing the formation of carbon filler network and percolation behaviour in the rubber composites.
[17]	2021	It was reported that the electrospinning processing method could be optimized to alter the relations between viscosity and nanofiber diameter across the whole range of copolymer concentrations and acetone participation. This emphasizes the critical influence of processing parameters on the materials preparation process.
[18]	2022	The processing parameters and material properties have impacted the electrical conductivity of solvent-based precursors derived from polymer composite.
[19]	2023	The study found that optimizing mixing speed and time could improve the acidity value of refined, bleached, used vegetable oils as a dielectric liquid in a transformer.

The table suggests a limited amount of research on the electrical aspect of SiR with micro-filler, despite the importance of processing parameters. Due to the focus on nano-fillers, researchers may have overlooked the analysis of the effects of different processing parameters on micro-filled polymeric insulators. Therefore, this paper aims to study the impact of varying processing parameters, namely mixing speed and time, on SiR filled with micro filler. The material performance is carried out via the inclined plane tracking (IPT) test, which will be further supported by Fourier transform infrared spectroscopy (FTIR). Leakage current value (LC) is used to depict the insulator's conditions, indicating the degree of degradation. It is also connected with the insulator's surface resistance, highlighting the capability of the insulator [20].

2. METHOD

This section comprises materials preparation and IPT test setup. The types of material used are explained. The details regarding the experimental design were also provided.

2.1. Materials preparation

High-temperature vulcanized (HTV) SiR was used together with a heat stabilizer. The filler was micro-alumina trihydrate (66% alumina and 34% water), while the curing agent was 98% active dicumyl peroxide (DCP). All materials were used as they were initially, without further purification. Each sample comprises 100 pphr of SiR, 50 pphr of ATH filler, 2 pphr of a heat stabilizer, and 1 pphr of DCP. A total of 12 samples (i.e., 3 samples for each combination depicted in Table 2) were prepared. The processing parameters were determined based on the previous research [15]. The materials were weighed, mixed using a Haake internal mixer, and then vulcanized at 170 °C and 50 bar for 10 minutes. Then, the samples underwent a 12-hour post-curing process in a forced air oven at 130 °C.

Table 2. The blending properties for all 12 samples

Sample	Mixing speed (rpm)	Mixing time (min)
1	70	5
2	70	10
3	40	10
4	40	5

2.2. IPT test setup

The IPT test is in accordance with the standard BS EN 60587. The test is used to evaluate the performance of insulating material against tracking and erosion via rapid aging by determining their tolerable LC values. The samples were tested via a constant tracking method at 3.5 kV for 6 hours. The LC data were recorded at every interval of 10 minutes. The LC data is collected via data acquisition (DAQ) equipment and stored on a computer via LabVIEW Software. The full IPT test setup can be referred from the reference [21].

3. RESULTS AND DISCUSSION

The LC data obtained from the 6-hour IPT experiment were divided into three stages. These stages were chosen to represent the first, intermediate, and last hours of the IPT test. The stages were as follows: the first stage (10-60 minutes), the second stage (160-210 minutes), and the third stage (310-360 minutes). There were 18 data points per stage (6 data for each sample). The LC data for each stage were analyzed by means of histogram plots based on the frequency of occurrences. The data is then discussed in subsections 3.1, 3.2, and 3.3. In subsection 3.4, the mean and standard error of the LC data for the last hour of the IPT test were calculated to verify the data since this stage consists of the most critical data of the IPT. Then, the results of this stage will be connected to the next subsection 3.5, which includes the FTIR results for all samples.

3.1. The first stage (10-60 minutes)

The first stage of data for all four samples was represented in a histogram, as shown in Figure 1. In the histogram, right-skewed data indicates positive skewness, resulting from a lower boundary in a data set (LC in a lower range of values). Skewed to the right also means a greater mean value than the median, while skewed to the left is vice versa. Bimodal represent two distinct peaks, not necessarily of the same height, but both have the most frequency of occurrence. Unimodal has only one peak with a mean value equal to the median, while multimodal has three or more peaks. First, the shape of the histogram was observed to see their type of skewness. If there is confusion, the mean and median value is used to determine the type of histogram.

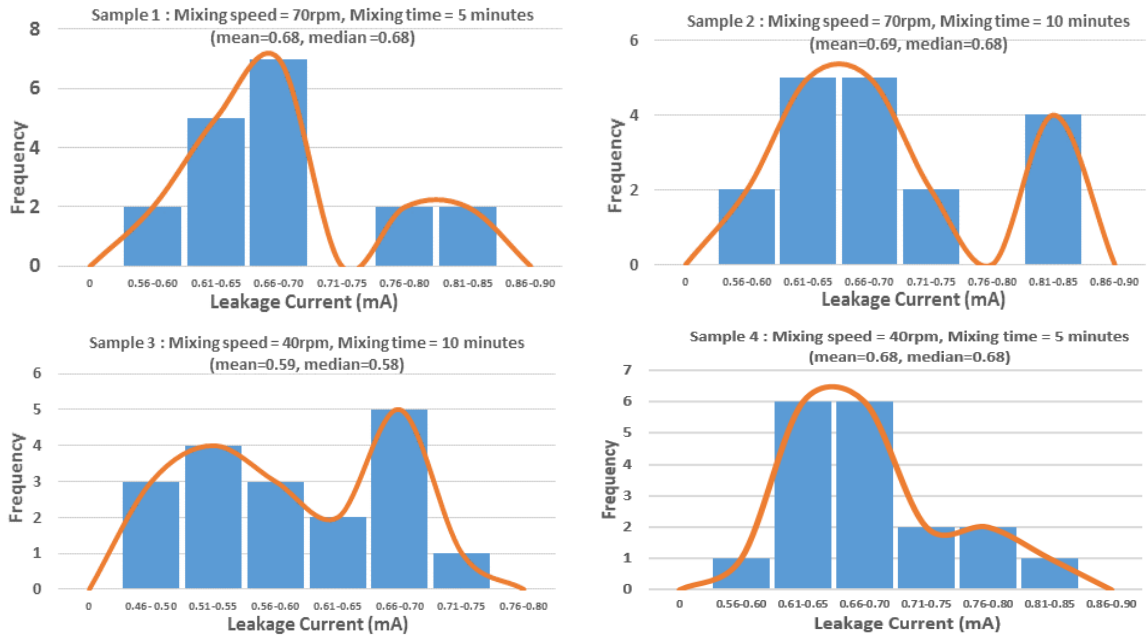


Figure 1. The LC trend for all samples in the first stage of the IPT test

In Figure 1, the LC readings for the first stage were primarily distributed in the range of 0.61 to 0.70 mA. Regarding the histogram shape, samples 1, 2, and 3 were bimodal with two distinct peaks, while sample 4 was skewed to the right. Despite the differences in processing parameters, the overall results showed a lower range of LC values. This is expected as LC values increase over time due to continuous wetting and heavy pollution, which leads to surface hydrophilicity and increased LC values [22]. Thus, more time is needed to observe any changes in trends in the LC range, which can be seen in the following two stages.

3.2. The second stage (160-210 minutes)

In the second stage, the LC range for all samples was plotted at the middle hours of the IPT test, which was 160 minutes to 210 minutes. Figure 2 shows the histogram for all samples at the second stage of the IPT test. An increase in the IPT time is usually followed by an increase in LC values. In this stage, the increase in LC values was proven when the mean values of all plots were slightly higher than those of stage 1 except for sample 2. The histogram shapes for samples 1 and 3 are multimodal. Sample 4 is skewed to the right, while sample 2 is symmetrical in shape. In terms of LC values, samples 3 and 4 had reached the LC ranges of 0.91-0.95 mA, slightly higher than samples 1 and 2. Sample 2, with the highest mixing speed and time, has a better mean than the other three samples. The filler dispersion is estimated to be better due to its processing parameters, which could be further confirmed in the next stage.

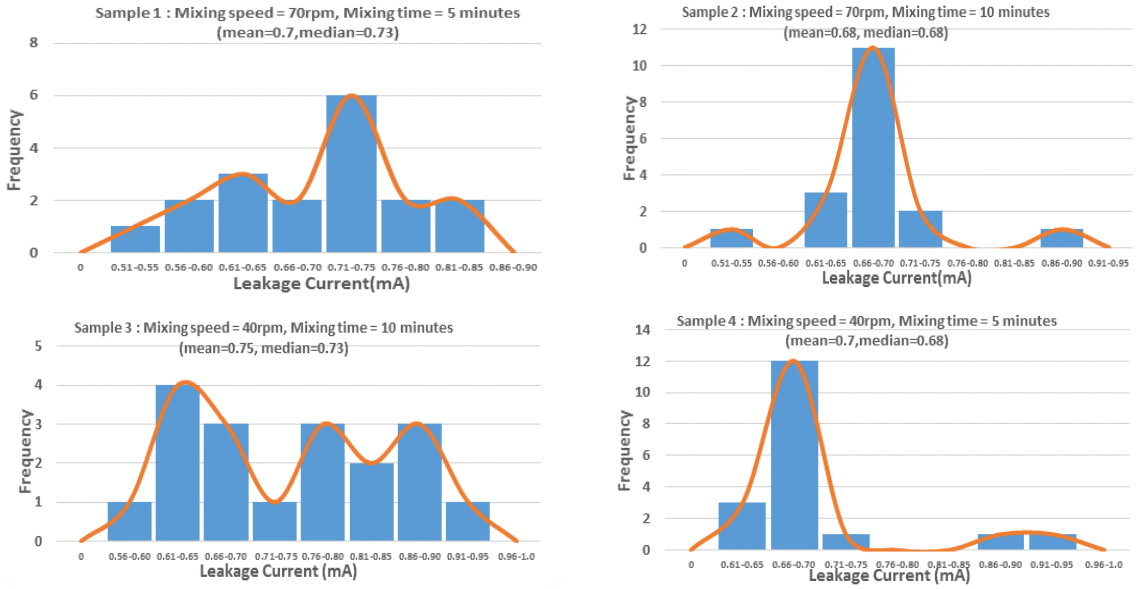


Figure 2. The LC trend for all samples at the second stage of the IPT test

By comparing samples 1 and 2 of higher mixing speeds (70 rpm), sample 2, with a higher mixing time of 10 minutes, seems to have lower LC values. On the other hand, by comparing samples 3 and 4 at lower mixing speeds (40 rpm), the same pattern is not detected as the mean and median for both samples are equal despite the differences in mixing time. In this stage, it can be observed that sample 2, with the highest mixing speed and mixing time, had better performance against tracking and erosion. On the flip side, it should be noted that the lowest mixing speed and time did not contribute to sample 4 having worse LC ranges. However, before making any conclusive statement on the LC trend, the LC results of the next stage should be considered.

3.3. The third stage (310-360 minutes)

The third stage of LC values recorded during the IPT test for all samples is plotted in Figure 3. This stage represents the last hour from the 6 hours test of IPT, which is usually the most critical stage. The recorded LC values during this period are usually used to categorize the resilience of the tested samples. In comparison to the previous second stage, there is an increase in the mean values for all samples, indicating increased degradation and aging. Over time, electrical discharges such as DBA, corona, and partial discharge will cause depolymerization, leading to embrittlement and hydrophilicity of the polymer, increasing the LC

values [23]. An increase in LC will lead to the development of insulator flashover [24]. However, a suitable processing parameter with optimum filler concentration could help to prolong the service time of the insulator.

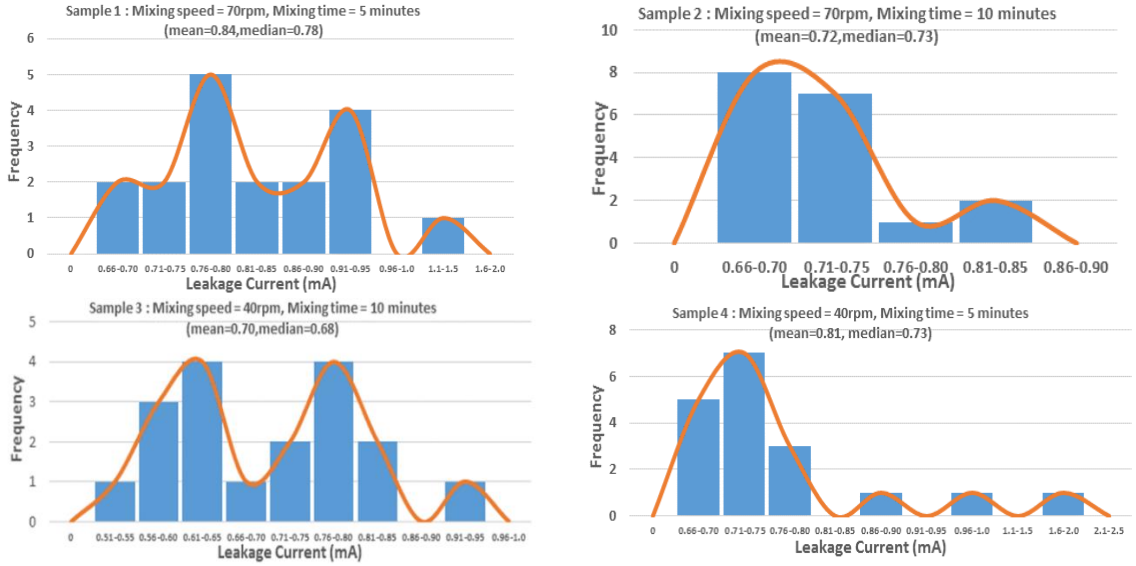


Figure 3. LC trend for all samples in the last hour of the IPT test

The histogram shape of samples 1 and 3 was a multimodal pattern, sample 2 was bimodal shape, and sample 4 was skewed to the right. In this stage, sample 2 has recorded the lowest mean value of 0.72 with a higher frequency of LC occurrences in lower ranges below 0.85 mA. Similar trends were also noticed in the previous stage, where the LC values for sample 2 were the lowest compared to other samples. The comparison of sample 1 and 2 of the same mixing speed (70rpm) showed that the one with 10 minutes of mixing time resulted in a better performance against tracking and erosion. Hence, in order to have better filler dispersion and higher resistance against tracking and erosion, 70 rpm and 10 minutes of processing parameters are suggested per 50 pphr of ATH filler concentration. A better dispersion rate provides better resistance against corona [25] and partial discharge due to less inter-particle distance (agglomeration) [26]. Similarly, for samples 3 and 4 with lower mixing speed (40 rpm), sample 3 with higher mixing time had a lower mean value. This emphasizes that mixing time is vital in ensuring that filler is appropriately dispersed.

A closer examination of the LC values trend for sample 2 reveals a higher frequency of occurrences in the lower range across all three tracking stages, distinguishing it from the other three samples. Next, interestingly, it was expected that sample 4 should have the highest mean value, but the highest mean was denoted by sample 1. This unpredictable twist again highlights the importance of mixing time, as 5 minutes could be insufficient for the filler to be dispersed properly even when the mixing speed was 70 rpm. However, it is worth noting that sample 4 exhibited a higher frequency of LC occurrences in the higher range, reaching up to 2 mA, which was the highest among all samples. It was stated that homogenous filler dispersion would significantly improve thermal stability and resistance against tracking and erosion [27]. Alternatively, increased agglomerations or impurities might reduce the space charge formation threshold of the composites, leading to a faster aging rate. Hence, regardless of the filler type, appropriate processing parameters are crucial for achieving homogeneous SiR composites with increased resistance to tracking and erosion.

3.4. Mean and standard error for the data of LC at the third stage

The data of LC from all samples for the last hour was analyzed via its mean and standard error values, which were calculated correspondingly using (1) and (2):

$$mean = \frac{sum\ of\ LC}{total\ number\ of\ LC\ observations} \quad (1)$$

$$standard\ error, \sigma_x = \frac{\sigma}{\sqrt{n}} \quad (2)$$

Where σ represents the standard deviation of LC data per sample, and n represents the total number of LC observations per sample, which is 18 in this case. The mean and standard error of the samples are plotted in

Figure 4. This analysis is crucial for assessing data stability and improving data representability. Sample 2 exhibited the lowest standard error of 1.18E-05, followed by samples 3, 1, and 4 with standard errors of 2.58E-05, 2.92E-05, and 7.36E-05, respectively. This further emphasizes the stability of the LC data for sample 2, likely due to the influence of processing parameters.

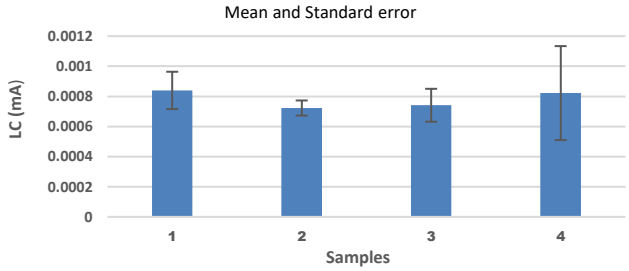


Figure 4. The standard error and mean for the LC data at the last hour of the IPT test

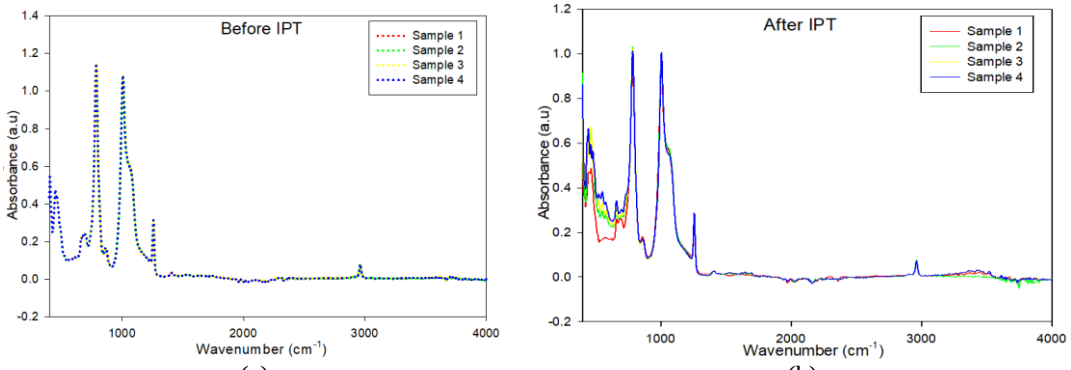
3.5. FTIR of SiR samples before and after IPT

FTIR spectroscopy is typically used to obtain absorption or emission of an infrared spectrum of a material. In this section, FTIR was used to inspect the structural changes in the SiR samples before and after the IPT test. The prominent groups and relevant absorption bands related to SiR samples are shown in Table 3. Figure 5 illustrates the FTIR spectra of all samples before and after IPT.

Figure 5(a) shows that all samples had identical absorbance spectra before the IPT test, despite being prepared with different processing parameters. However, sample discrepancies can be observed after the IPT test in Figure 5(b). Next, Figure 6 depicts the important magnified peaks of the absorbance spectrum, representing the functional groups. All the peaks exhibit a consistent pattern of reduced absorbance values after the IPT test. The reduction in absorbance value was due to the depolymerization of SiR [28], [29].

Table 3. Relevant absorption band for SiR samples

Functional group	Wavenumber (cm^{-1})
Silicone oxygen stretching (O-Si$(CH_3)_2$-O)	795-850
Siloxane stretching (Si-O-Si)	1010-1080
Silicone methyl stretching (Si-CH_3)	1240-1270
Carbon-hydrogen stretching (CH_3)	2963-2950
Hydroxyl hydrophilic (OH in ATH)	3200-3700



(a) (b)

Figure 5. Absorbance peak for all samples; (a) before IPT and (b) after IPT

In Figure 6(a), sample 2 has the lowest reduction in the O-Si$(CH_3)_2$-O bond, followed by sample 3, sample 4, and sample 1. The difference in the absorbance values was not large but noticeable. The results align with the increasing mean values of the samples in subsection 3.3. In Figure 6(b), the absorbance line for sample 2, corresponding to the Si-O-Si bond, consistently exhibits a higher value than the other three lines. This indicates the lowest reduction in absorbance peak among the four samples. The absorption peak reduction

for sample 2 is 7.57%. This reduction suggests degradation, specifically the depolymerization of the Si-O-Si backbone chain in SiR. Previous research has shown varying loss percentages in absorbance peaks based on filler types and concentrations. For instance, in a sample of micro ATH and nano silica, a 13.26% loss of the Si-O-Si group was reported after rapid aging [30]. Additionally, two SiR samples filled with different micro and nano-sized ATH concentrations reported 27.2% and 31.5% reductions in absorbance peak, respectively [31]. This suggests that modifying processing parameters may be more effective in preventing aging as sample 2 exhibits a significantly lower reduction compared to the previous study. Figure 6(c) shows slight differences in the absorbance peak among the four samples, with sample 2 slightly higher at the peak. Similarly, in Figure 6(d), the carbon-hydrogen stretching side chain exhibits the lowest reduction in sample 2, with sample 1 showing the most reduction in the absorbance peak. During IPT, the polymer experiences chain scissoring or even further crosslinking due to oxidation, hydrolysis, hydrosilylation or pyrolysis, causing the absorbance peak of the SiR functional group to reduce. Adding filler helps in increasing the materials resistibility against aging as the linking between the base polymer matrix and filler can results in the alterations of the bulk properties of the composite insulating materials [32], [33]. Hence, it can be inferred that the chosen processing parameters in samples 1, 3, and 4 might not be optimal for achieving proper filler dispersion, resulting in reduced functional groups and accelerated aging of the samples.

Alternatively, the trend differs for the OH group shown in Figure 6(e). The lowest absorbance was recorded by sample 2. Hydroxylation occurs after aging, and the presence of hydroxyl indicates the aging and hydrophilicity of the sample [29]. Despite the similar filler content, the difference in mixing parameters resulted in a better performance of sample 2 against the tracking and erosion.

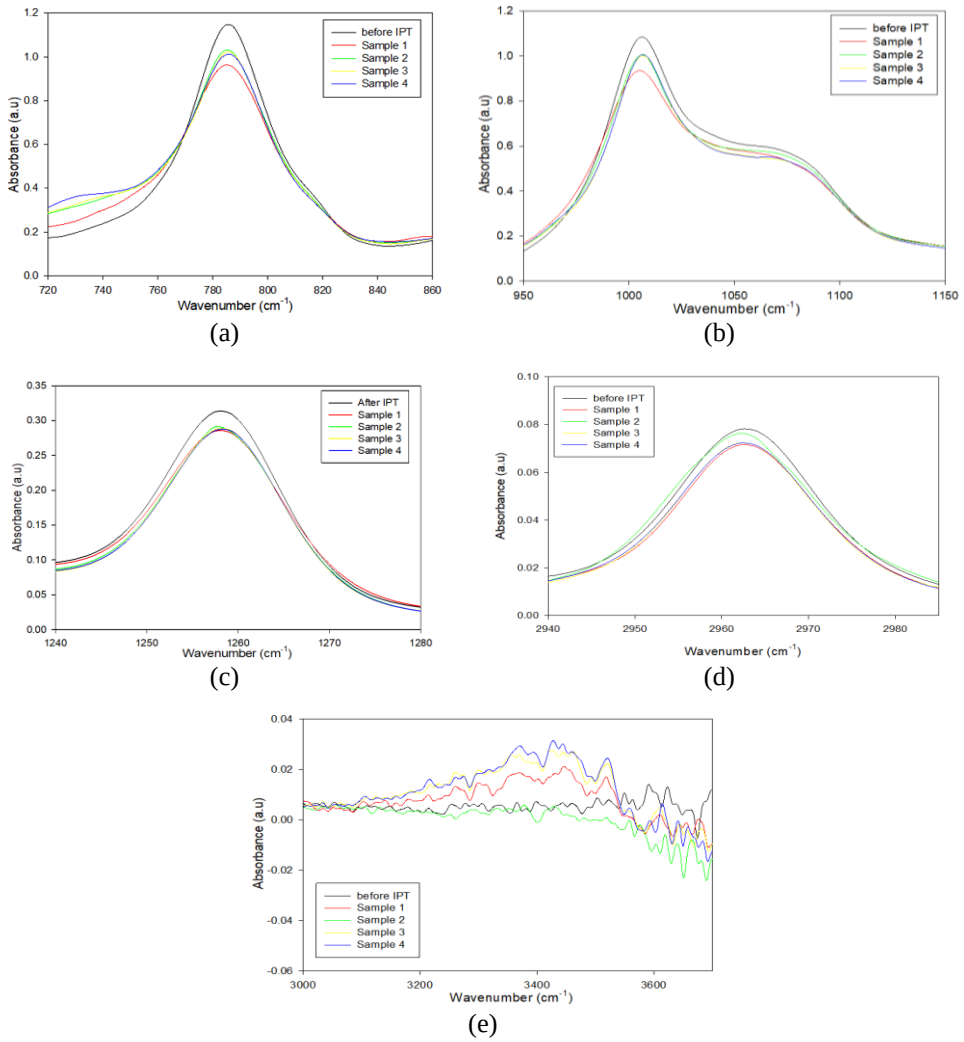


Figure 6. Magnified relevant absorption peaks for all samples of; (a) silicone oxygen stretching, (b) siloxane stretching, (c) silicone methyl stretching, (d) carbon-hydrogen stretching, and (e) hydroxyl hydrophilic

4. CONCLUSION

The analysis of processing parameters' effect on LC values of SiR with micro-filled ATH has revealed several findings. Initially, during the early stage of the IPT test, no significant differences were observed in the frequency and patterns of LC among the samples. In the second stage (160-210 minutes), the samples exhibited varying LC patterns, with sample 2 showing the lowest mean value. In the final hour of the IPT test, sample 2, which had the highest mixing speed and time, recorded the lowest mean LC values ranging from 0.66 to 0.85 mA. FTIR results indicated that sample 2 displayed the best resistance against tracking and erosion. Although the differences among the samples were relatively small, the impact of processing parameters was highlighted. The relationship between processing parameters and their influence on LC ranges and the performance of samples against tracking and erosion is evident. In future, further studies on the processing of polymeric insulators with additional testing should be done to enhance HV insulation.

ACKNOWLEDGEMENTS

The authors wish to extend their utmost appreciation to the Universiti Teknikal Malaysia Melaka (UTeM) for the support of facilities provided during the research. An expression of gratitude is also given to Immortal Green Industrial Sdn. Bhd. for the research materials support.

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