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Preparation of Insulating Materials Based on Natural Rubber/Polyethylene Blend and Nanosilica

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Abstract

In the present work, a preparation of insulating materials based on polyethylene/natural rubber/nanosilica (PE/NR/nanosilica) blend was investigated. The optimal condition and composition for the blends were determined including types of natural rubber (i.e. RSS, SVR3L and deproteinized natural rubber-DPNR), type of nanosilica (silica_M with specific surface area of 340-420 m^2/g and silica_255G with specific surface area 285 m^2/g), blending methods and conditions. The mechanical properties, electrical insulation properties and thermal stability of the blends were characterized by tensile test, dielectric strength and thermal gravimetric analysis for each condition. It was found that the mass ratios of PE (L21 type) and NR of 20/80 and a silica content of 4% will result in a good mechanical and dielectric strength, which was 13.2 MPa and 100 kV/mm, respectively. RSS type and silica_M showed good compatibility with L21 in the blend at optimal conditions for the blend preparation. This result was further approved by SEM analysis and FTIR spectroscopy. The blend sample, L21/RSS@silica_M was exhibited better thermal stability and dielectric strength than L21/SVR3L@silica_M and L21/DPNR@silica_M samples and it demonstrated that L21/RSS@silica_M blend samples may be appropriate for electrical insulation application.

Keywords: Insulating materials, natural rubber, polyethylene, silica, dielectric strength.

1. Introduction

Thermoplastic elastomers (TPEs) are selected for electrical insulation applications due to their superior performance and cost-effectiveness. These materials exhibit high mechanical strength, excellent electromechanical connectivity, and significant resistance to moisture absorption [1]. In previous studies, polypropylene (PP)-based TPEs have drawn significant research interest for their potential to replace crosslinked polyethylene in direct current cable insulation [2-6].

Polyethylene (PE) is a widely used insulating material for electrical cables due to its low electrical conductivity, high dielectric strength, and low dielectric loss at high frequencies. With its superior properties, PE has replaced traditional insulating materials such as styrene-butadiene rubber, chloroprene rubber, and ethylene propylene diene monomer (EPDM), while also making cables lighter and more compact. Currently, the high-voltage (HV) cable industry is shifting from Low Density Polyethylene (LDPE) to Linear Low Density Polyethylene (LLDPE) due to LLDPE's higher environmental resistance and flexibility [7].

Additionally, numerous researchers have investigated the electrical properties of PE-based TPEs for electrical insulation applications [8-14]. Makmud *et al*. [15] and Arief *et al*. [16] analyzed the insulation performance of LLDPE/natural rubber (LLDPE/NR) under high voltage and found that variations in blend components affected their response to electric fields, thereby influencing partial discharge behavior. Jamail *et al*. [17] evaluated the leakage current of LLDPE/NR blends with varying concentrations of nanosilica and titania fillers, revealing that the presence of nanofillers enhanced electrical tracking resistance, with the 1 wt% titania sample exhibiting the lowest leakage current and the highest resistance to electrical tracking. Despite the addition of NR, a sustainable and environmentally friendly material, research on thermoplastic elastomers derived from deproteinized natural rubber (DPNR) and LLDPE for insulation, labor protection, and electrical safety applications is still limited.

Natural rubber, either commercial grades, i.e., Standard Vietnam Rubber 3L (SVR3L), Ribbed Smoked Sheet (RSS) or DPNR, is well known for excellent elasticity and impact resistance added to LLDPE, which may enhance thermal stability and processability for the blend between them. Furthermore, nanofillers such as nano silica also improve mechanical and electrical properties provided it disperses uniformly. In the present study, we

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investigated how various types of rubber, silica content, and processing conditions affect LLDPE/NR blend properties. It would have provided us with a better understanding of the compromise between the fillers, types of rubbers, processing conditions onto the properties of the blends, and dielectric strength of the blends. We also observed the morphology of the blends to explore valuable insights for designing high-performance materials with superior mechanical strength and electrical insulation.

2. Experiments

2.1. Materials

The materials used in this study were SVR3L (from Phuoc-Hoa Company, Vietnam), RSS (from Mega Company, Vietnam), DPNR prepared from high ammonia natural rubber (HANR) with a dry rubber content (DRC) of about 60%. LLDPE 21HS plastic (designated as L21), from Qamar, Saudi Arabia with density 0.918 g/cm^3, melt flow index (MFI) of 2g/10 min. Nanosilica (designated as silica_M) from Merck KGaA, Germany (surface area of 370 - 420 m^2/g), and Tokusil 255G nanosilica (designated as silica_255G) from Thailand (surface area of 285 m^2/g).

2.2. Blend Preparation and Sheet Molding

Preparation of blend

The PE/NR blends were melt-compound using an internal mixer at a rotation speed of 18 rpm. First, the mixer was heated to the required temperature for blend preparation, and the PE was added and mixed until it melted uniformly for 2 minutes. Then, the rubber was added, and the mixture was blended for another 10 minutes.

The PE/NR/nanosilica was prepared with two different procedures.

Procedure 1: Nanosilica was first mixed with PE at a melting temperature of PE for 6 minutes before mixing with NR. The samples were denoted as PE@silica/NR blends.

Procedure 2: Nanosilica was first mixed with NR at 60 ^{o}C and maintained for 6 minutes, after that the resulting mixture was mixed with PE. The samples were abbreviated as PE/NR@silica blends.

The PE/silica blend sample was prepared as follows. First, silica was added to L21 plastic by heating at 130 ^{o}C for the L21 to flow constantly for 2 minutes, then silica was added and continued to mix for further 7 minutes before it was pressed into a thin film for characterization.

Molding conditions

After blending, the mixtures were compressed into rubber sheets at 130 ^{o}C for 10 minutes under a pressure of 10 MPa and then cooled to room temperature. The thickness of the sheet was approximately 0.2 mm.

2.3. Characterizations

The tensile strength and elongation at break of the blends were determined using an Instron universal testing machine according to ASTM D-638 standard. The measurement was performed at room temperature and was repeated 5 times.

The dielectric strength of the samples was determined using the Sefelec Dielectric Withstand Tester according to ASTM D149 standard. The sample was cut into dimensions of 100 mm x 100 mm, with clean surfaces which were free from defects before testing. The thickness of the samples was controlled ranging from 0.1 mm to 1 mm. At least five samples of each type were tested.

The morphology of the samples was investigated using SEM (JEOL JSM-7600F). Before the observation, the samples were cut into small pieces and immersed in liquid nitrogen for some time and then cryo-facture was performed to obtain the clean cross-section surface. A thin conductive coating, such as gold, was then coated to get a better image. The gold-coated samples were placed in the SEM vacuum chamber, and the condition was adjusted before capturing the images of the material's internal structure and surface morphology.

The chemical structure and the presence of non-rubber components such as proteins in NR were characterized with an attenuated total reflectance (ATR) using Nicolet TM iS20 FTIR spectrometer from 400 to 4000 cm^{-1}, resolution of 4 cm^{-1} and 32 scans. Thermal gravimetric analysis (TGA) of the blend was performed with NETZSCH STA 449F5 under atmospheric conditions from 25 ^{o}C to 600 ^{o}C, with a scanning rate of 10 ^{o}C/min. About 10 mg of the blend samples packed in a platinum pan were used for TGA measurement.

3. Result and Discussion

3.1. Effect of L21/RSS Mass Ratio

In the present work, three compositions of PE/NR blends, which were 75/25, 80/20, and 85/15 mass ratios of PE and NR were investigated. These three PE/NR blends were characterized by tensile strength and dielectric strength to find the optimum PE/NR ratio.

Fig. 1 shows the stress-elongation curve of L21/RSS blend with three different compositions of L21 and RSS of 75/25, 80/20, and 85/15. As can be seen, when rubber content increased from 15% (in L21/RSS 85/15) to 20% (in L21/RSS 80/20) and to 25% (in L21/RSS 75/25), the modulus of the blend slightly decreased.

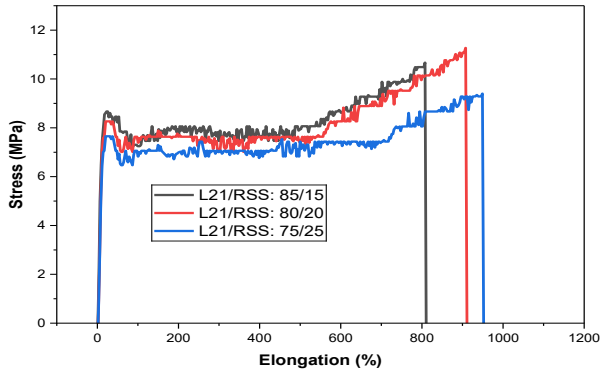

Fig. 1. Stress-elongation curves for L21/RSS blend at compositions of 75/25, 80/20, and 85/15

In particular, the sample with the lowest rubber content (L21/RSS 85/15) exhibited the highest modulus, while the sample with 25% rubber content (L21/RSS 75/25) showed the lowest modulus. This is because unvulcanized natural rubber has low hardness and strength. Therefore, increasing its content in the blend results in a reduction in the blend's strength. It was noted that the sample L21/RSS with 20% content of rubber showed the highest tensile strength and elongation at break among three samples. It indicated that the optimum rubber content in the blend was determined to be about 20%. This optimum content of rubber in the blend would be used in further investigation.

3.2. Effect of Silica Content in the Blend

The role of nanosilica in the properties of the PE/NR/silica blend was investigated in the present work with silica content varying from 2%, 4%, and 6%. The silica_M was used in this investigation and the preparation for PE/NR/silica blend was performed with the procedure 2. Fig. 2 shows the stress-elongation curves for L21/RSS@silica blend.

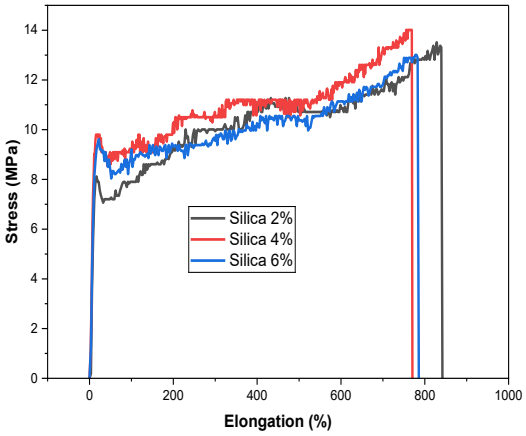

Fig. 2. Stress-strain curves of L21/RSS@silicaM blend at silica contents of 2%, 4% and 6%

The result from Fig. 2 revealed that increasing the silica content from 2% to 4% resulted in improvements in tensile strength and elongation at break, however, when the nanosilica content was increased to 6%, a decrease in tensile strength was observed. The sample with 2% nanosilica exhibited the lowest strength, indicating that 2% nanosilica was insufficient to significantly improve the mechanical properties due to ineffective reinforcement and uneven dispersion. Meanwhile, the sample with 4% nanosilica achieved the highest tensile strength and modulus, suggesting that 4% nanosilica content was the optimal amount for nanosilica to be dispersed uniformly and it resulted in effective reinforcement for the blend. At 6% nanosilica the stress-elongation curve showed a decrease in modulus compared to the sample with 4% silica content, although it was slightly higher than that of the blend sample with 2% silica content. This may be resulted from too much silica content that exceeded the critical dispersion threshold, causing the agglomeration of nanosilica particles and subsequently reducing the effectiveness of the reinforcement. Therefore, an optimum nanosilica content was found to be 4% and this value was used in further investigation.

3.3. Effect of Nanosilica Type

The effect of nanosilica type on the preparation of the blend was investigated. Two types of nanosilica with different surface areas were used in this study, with the same silica content of 4%.

Table 1. Electric strength of samples using different types of nanosilica

Sample	Electric strength (kV/mm)
L21	41.7
L21@silica_255G	45.5
L21@silica_M	65.0

Table 1 shows the dielectric strength for the L21, L21@silica_255G, and L21@silica_M blends. The dielectric strength of the L21@silica_255G and L21@silica_M blends was higher than that of the L21 sample. This indicates that the addition of nanosilica improved voltage endurance for the L21 sample. Specifically, the dielectric strength increased with the use of silica_M and silica_255G compared to the L21 sample. Among these, the L21@silica_M blend exhibited the highest voltage endurance, showing a 56% improvement over the original L21 sample.

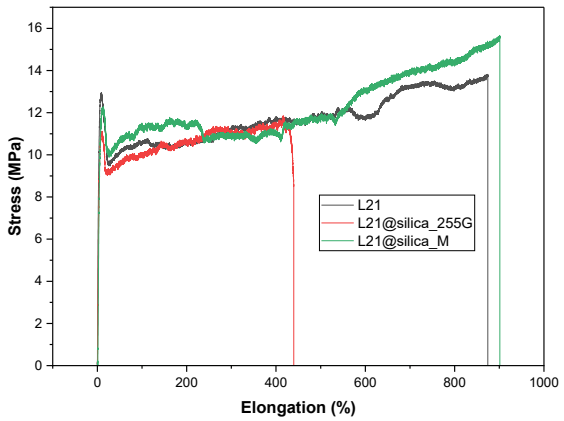

Fig. 3. Stress-elongation curves of L21, L21@silica_255G and L21@silica_M blends

The mechanical properties of the blends were also investigated. Fig. 3 shows mechanical properties for L21, L21@silica_255G, and L21@silica_M blends. The elongation at break of L21@255G was at more than 450%, which was lower than elongation at break of L21 (880%) and L21@silica_M (900%). Furthermore, the stress at break of L21@silica_M blend also reached the highest value, at 15.5 MPa. This value is slightly higher than stress at break of L21 (13 MPa) and much higher than that of L21@silica_255G (11.5 MPa). From these results, it may be demonstrate that the incorporation of silica_255G has a negative effect on the strength of the blend. It may be because the silica_255G has poor interaction with PE matrix due to the low surface area. The silica particles may be too big to incorporate with PE. In contrast, the blend L21@silica_M, which uses nanosilica with a higher surface area, shows a better reinforcement effect of nanosilica in the mechanical properties of the blend. Thus, the surface area of nanosilica has an important effect on the improvement of mechanical properties of the PE/silica blends.

It was observed that the method used to incorporate nanosilica may have influenced the mechanical properties of the blends. In this study, we investigated two different procedures for preparing PE/NR/silica blends, as described in the experiment section. Fig. 4 shows the mechanical properties of L21@silica_M/NR and L21/NR@silica_M, which were prepared using procedure 1 and procedure 2, respectively. The L21/RSS ratio was 80/20, and 4% silica_M was used in both cases.

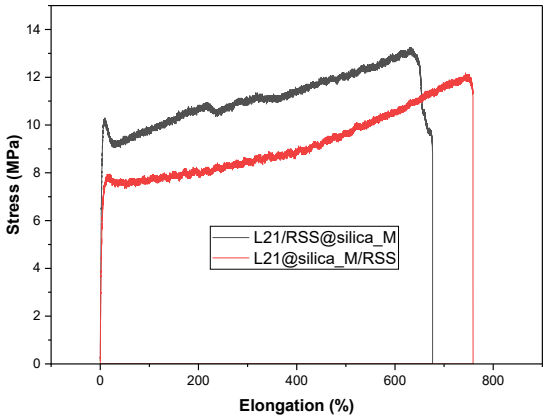

Fig. 4. Stress-elongation curves of L21/RSS @ silica_M and L21@silica_M/RSS blends

As shown in Fig. 4, the sample L21/RSS@silica_M exhibited better mechanical performance than L21@silica_M/RSS. Specifically, L21/RSS@silica_M demonstrated higher stress at break and a higher modulus compared to L21@silica_M/RSS. This result indicated that incorporating nanosilica into rubber before mixing with PE leads to a more significant reinforcement than incorporating nanosilica into PE before mixing with NR. This improved performance may be attribute to better compatibility between nanosilica and rubber, rather than between nanosilica and PE. The presence of non-rubber components, such as proteins in RSS, may also enhance the compatibility between nanosilica and natural rubber.

Table 2. Dielectric strength of the blends prepared by different procedures

Sample name	**Dielectric strength (kV/mm)**
L21@silica_M/RSS	73
L21/RSS@silica_M	100

Table 2 presents dielectric strength values for L21@silica_M/RSS and L21/RSS@silica_M. Interestingly, the sample L21/RSS@silica_M showed a higher dielectric strength of 100 kV/mm, compared to 73 kV/mm for L21@silica_M/RSS. This suggested that the dispersion of nanosilica in the blends influences the dielectric strength. The improved dielectric strength in the L21/RSS@silica_M sample may be due to better dispersion of nanosilica in the blends prepared using procedure 2.

3.4. Effect of Temperature on the Preparation of PE/NR silica Blends

Temperature is a critical factor in melt blending. In this study, we investigated the preparation of the blends at three different temperatures: 130 °C, 150 °C, and 170 °C. The blends were prepared using procedure 2 and their mechanical properties are shown in Fig. 5. It was observed that increasing the temperature led to a decrease in the tensile strength. Specifically, as the mixing temperature rose from 130 °C to 150 °C and 170 °C, the tensile strength gradually declined. The blend prepared at 130 °C exhibited the highest stress at break, reaching approximately 15 MPa, while the blend prepared at 150 °C reached around 13 MPa, and the blend prepared at 170 °C showed the lowest stress at break, at about 10 MPa. The inverse relationship between processing temperature and mechanical strength may indicate thermal degradation at higher temperatures. Thus, the optimum temperature for blend preparation is about 130 °C.

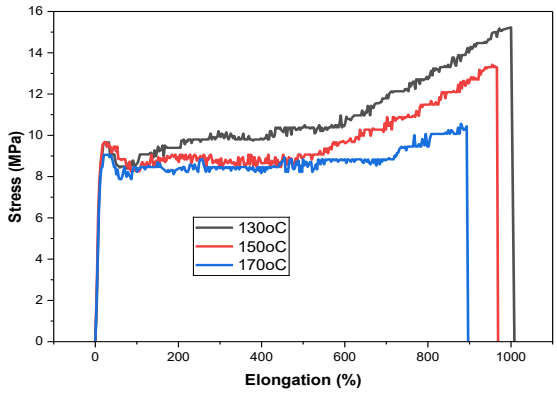

Fig. 5. Stress-elongation curves of L21/RSS@ silica_M blends processed at different mixing temperatures

3.5. Effect of Different Types of Rubber

To select a suitable type of NR for blending with PE and nanosilica, three NR types were used in this study. The mass ratio between NR and PE was kept constant at 80/20, and 4% silica_M was incorporated in each blend. All blends were prepared using procedure 2.

Fig. 6 shows IR spectra for SVR3L, RSS and DPNR. The vibration at nearly 3000 cm^{-1} was due to vibration of C-H bonds. The characteristic adsorption band at 1660 cm^{-1} was assigned to vibration of C=C bond in NR structure. The small signal at 3282 cm^{-1} and 1552 cm^{-1} were due to the presence of proteins.

The intensities of these bands for DPNR were quite lower than those for RSS and SVR3L. This suggested that the amount of protein in DPNR is negligible compared to those in RSS and SVR3L. The presence of proteins in RSS and SVR3L may contribute to the dispersion of nanosilica and the compatibility of NR with PE and nanosilica during the preparation of the blend.

Table 3. Electrical strength of blend samples using different types of rubber

Sample name	Dielectric strength (kV/mm)
L21/DPNR@silica_M	76
L21/RSS@silica_M	100
L21/SVR3L@silica_M	66.7

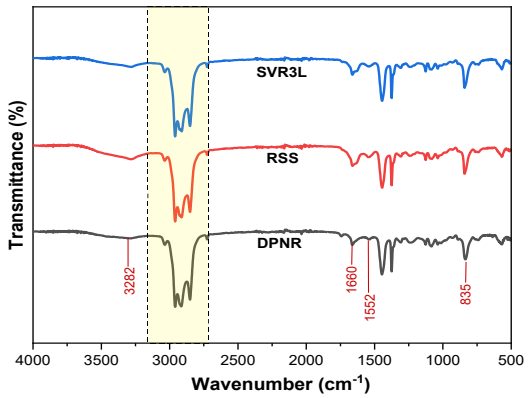

Fig. 6. FTIR spectra for SVR3L, RSS and DPNR

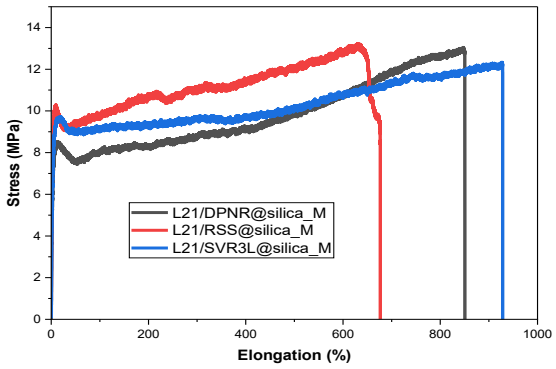

Fig. 7. Stress elongation curves of L21/DPNR@Silica_M, L21/RSS@silica_M, 21/SVR3L@silica_M

Fig. 7 presents the stress-elongation curves for the L21/DPNR@silica_M, L21/RSS@silica_M, and L21/SVR3L@silica_M. The results show that the L21/RSS@silica_M blend exhibited the highest modulus, with a stress at break of 13.2 MPa, and an elongation at break of 700%. In contrast, the L21/DPNR@silica_M blend displayed the lowest tensile strength, with a stress at break of 12.9 MPa and an elongation at break of 850%. The L21/SVR3L@silica_M has a tensile strength of 12.4 MPa and the highest elongation at break among

the three, reaching over 900%. The difference in mechanical properties of the blends concerning three types of NR may be due to the interaction of the silica with NR.

Table 4. Electrical strength of blend samples using different types of rubber

Sample name	Dielectric strength (kV/mm)
L21/DPNR@silica_M	76
L21/RSS@silica_M	100
L21/SVR3L@silica_M	66.7

The breakdown electrical strengths of the blends are shown in Table 4. The L21/RSS@silica_M exhibits the highest electrical strength compared to the other two blends. The dielectric strength of L21/RSS@silica_M is approximately 100 kV/mm, which is significantly higher than that of L21/DPNR@silica_M (76 kV/mm) and L21/SVR3L@silica_M (66.7 kV/mm). Based on these results, we can conclude that the L21/RSS@silica_M not only has the highest mechanical properties but also the highest electrical strength. This blend meets the requirement for insulating materials due to its combination of high mechanical and electric strengths.

Fig. 8 illustrates the TGA and dTGA curves for L21/DPNR@silica_M, L21/RSS@silica_M, and L21/SVR3L@silica_M. The decomposition of the blends occurred in two steps. The first step has the highest TGA peak at 360 ºC. This step may be due to the decomposition of PE. The second step had the highest dTGA peak at around 500 ºC and it was ascribed to the decomposition of NR.

The mass remained unchanged after 520 ºC and the residue ash content was about 4-5%, which was due to silica incorporated in the blends. It was also noted that, the L21/DPNR@silica_M started to decompose earlier at a faster rate than the other two samples. This may conclude that L21/RSS@silica_M and L21/SVR3L@silica_M could exhibit better thermal stability than L21/DPNR@silica_M.

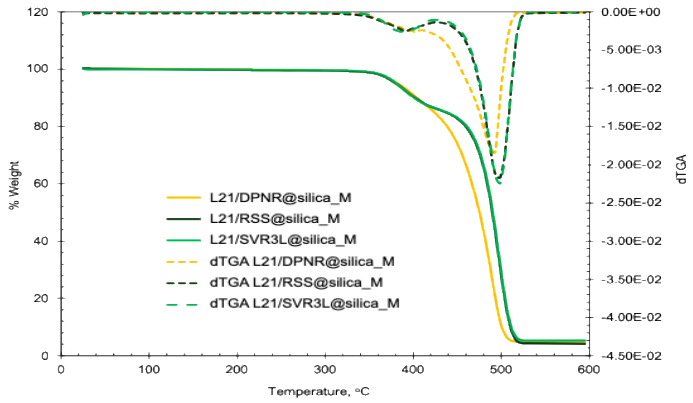

Fig. 8. TGA and dTGA curves of L21/DPNR@silica_M, L21/RSS@silica_M, and L21/SVR3L@silica_M

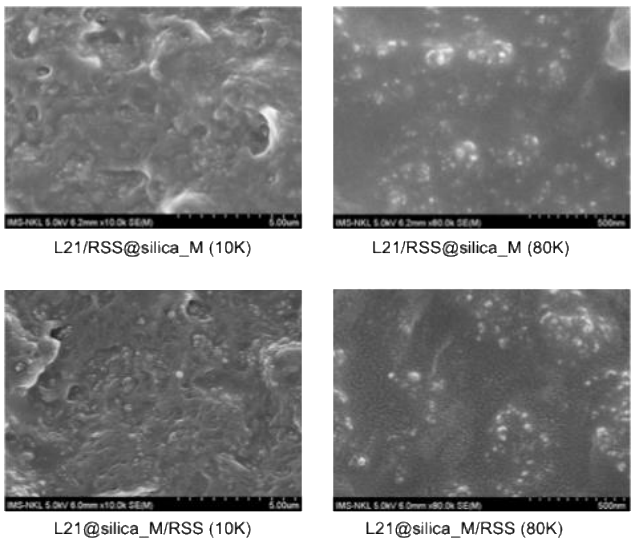

Fig. 9. SEM micrographs of L21/RSS@silica_M and L21@silica_M/RSS at 10K and 80K magnification

3.6. Morphology Observation

Fig. 9 shows the SEM images of the L21/RSS@silica_M and L21@silica_M/RSS blends at 10,000x and 80,000x magnification. From the SEM images, it is possible to see the distribution of nanosilica in the blends using two procedures. At 10,000x magnification, it can be observed that nanosilica disperses more uniformly in the L21/RSS@silica_M compared to the L21@silica_M/RSS. The L21/RSS@silica_M blend shows more uniform nanosilica domain sizes. This observation suggests that nanosilica has better compatibility with the NR than with PE. When nanosilica is mixed with NR first, followed by PE, it disperses more effectively in the NR phase. In contrast, when nanosilica is mixed with PE first, then NR, it leads to inhomogeneous (nonuniform or uneven) dispersion. At 80,000x magnification, significant differences in nanosilica dispersion were observed between the two blending methods.

The SEM image of the L21@silica_M/RSS blend showed that nanosilica tended to cluster into aggregates, while the SEM image of the L21/RSS@silica_M blend revealed a smoother structure, with smaller silica particles more evenly distributed. The uniform distribution of nanosilica in the L21/RSS@silica_M blend likely contributed to the enhanced mechanical properties of the blend, as further supported by the tensile strength curves discussed above. These observations indicated that pre-incorporating nanosilica into the rubber phase resulted in better dispersion characteristics, likely due to natural rubber's better compatibility with nanosilica. The sequence of incorporation method had a significant impact on the morphology of the blend.

4. Conclusion

In this study, we successfully investigated and optimized the preparation of polyethylene/natural rubber blend with nanosilica. The optimal blend composition was found to be 80/20 mass ratios of PE/NR and 4% of nanosilica. We also explored different methods for incorporating nanosilica into the blends. It was found that incorporating silica into NR phase followed by mixing with PE has resulted in better dispersion of nanosilica in the blends, thereby enhancing both mechanical properties and electrical properties.

The suitable temperature for blending was 130 ^{o}C. Among three natural rubbers used, RSS was found to yield the best mechanical and electrical performance with tensile strength of 13.2 MPa and a dielectric strength of 100 kV/mm. This conclusion was further supported by SEM images, which showed a more uniform dispersion of nanosilica in L21/RSS@silica_M blend. These findings provide valuable insights into the development of high-performance PE/NR blends with enhanced mechanical and electrical properties. The optimized processing conditions and material combinations established in this study can serve as a foundation for industrial applications that require both mechanical durability and electrical insulation properties.

Acknowledgements

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