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บทความวิจัย

Impact of Cu-modified Activated Carbon on Natural Rubber Sheet's Mechanical Properties

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Abstract: This investigation evaluated the results of incorporating copper-modified activated carbon into natural rubber sheets on their electrical and mechanical characteristics. copper-modified activated carbon was added at various concentrations (5, 10, and 15 parts per hundred rubber), resulting in a notable enhancement in density as confirmed by scanning electron microscopy (SEM). However, the addition of copper-modified activated carbon led to a deterioration in several mechanical properties, including hardness, tensile strength, elongation at break, and rip strength, with the most significant decline observed in tensile strength. Atomic force microscopy (AFM) analysis revealed that copper-modified activated carbon addition in natural rubber sheets exhibited enhanced electrical properties compared to those containing only activated carbon. The findings suggest that these rubber sheets offer a promising balance between dielectric constants and mechanical durability, making them potential candidates for applications demanding flexible sensors and electrostatic discharge protection.

Keywords: Natural rubber, Cu-modified, activated carbon, tensile strength

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1. Introduction

The electronics industry faces significant challenges due to static electricity, primarily caused by the buildup of electrostatic discharge (ESD). ESD can damage integrated circuits, disrupt control systems, and increase manufacturing costs [1, 2]. To combat this, ESD protection systems are used, often incorporating anti-static materials to prevent the buildup of static electricity and protect components and equipment. Rubber plays a key role in these systems, offering both flexibilities to cushion delicate parts and durability for robust surfaces [3, 4]. This study explores the use of rubber in ESD protection, emphasizing its unique balance of softness and strength in various applications.

Advancements in processing methods and the addition of various components have allowed natural rubber to be adapted to meet the specific needs of different industries. Carbon black (CB) powder, known for enhancing rubber properties, is one of the most commonly used additives. It enables manufacturers to customize the performance of rubber products [5]. When CB is added to rubber sheets in amounts greater than 15-30 phr, it creates conductive sheets. This conductivity comes from the formation of a conductive network within the rubber, where the increased filler content reduces the gaps, allowing electrons to move more easily and improving overall conductivity.

Several factors affect electrical conduction, including temperature and the properties of the filler material. As temperature increases, thermal expansion causes the polymer molecules and the conductive network to expand, resulting in a drop in conductivity. The size and surface area of the filler particles also play a crucial role in conductivity [6]. Evenly dispersing carbon particles throughout the rubber matrix significantly improves electrical conductivity compared to when the particles are clustered. Adding carbon nanotubes further enhances the conductivity by increasing the efficiency of the carbon particle network [7].

Activated carbon (Ac), known for its large surface area, is widely used in applications like water purification and air filtration [8]. To produce charcoal powder, the charcoal undergoes physical or chemical activation, which alters its surface to increase its surface area, making it more effective for different reactions [9, 10]. Therefore, this study explores the incorporation of copper-modified Ac into rubber sheets. The Cu-modified Ac is prepared through chemical solution reduction, with copper concentrations of 1% and 2% by weight. Rubber sheets containing 5 phr, 10 phr, and 15 phr of filler are then prepared and analyzed using SEM-EDX to examine the structure and distribution of Ac and

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copper particles. The analysis focuses on how Cu modification affects the electrical conductivity of rubber sheets, along with its mechanical properties such as density, hardness, tensile strength, and elongation at break. The volume resistivity of the sheets is also measured. The aim is to understand how combining Ac with copper doping enhances both the structural and electrical properties of natural rubber composites.

2. Materials

In this study, natural rubber (STR5L) was selected as the polymer matrix, with a fixed concentration of 100 parts per hundred rubber (phr). The material was sourced from FAOT in Nakhon Sri Thammarat, Thailand, ensuring consistency in the base polymer used for experimentation. To facilitate the vulcanization process, stearic acid was incorporated as an activator at 1 phr, obtained from R.M.C. Supply Co., Ltd. in Nonthaburi, Thailand. Additionally, zinc oxide (ZnO), another essential activator, was introduced at 5 phr to enhance the crosslinking process, with the material supplied by Thai Lysaght Co., Ltd. in Pranakorn Si Ayutthaya, Thailand. To further optimize the rubber formulation, 2,2'-Dithiobis-(benzothiazole) (MBTs) was used as an accelerator at a concentration of 1 phr, procured from Siam Chemicals Co., Ltd. in Samutprakarn, Thailand. Sulfur (S) acting as a crucial role as a vulcanizing agent by promoting crosslink formation within the polymer network, was included at 2.5 phr, also provided by Siam Chemicals Co., Ltd. To modify and enhance the dielectric properties of the material, activated carbon (Ac) was introduced as a reinforcing filler at varying concentrations of 0 and 15 phr, with the material sourced from Carbokarn Co., Ltd. Furthermore, to further improve the electrical performance, copper was incorporated onto the activated carbon surface in weight percentages of 0, 1, and 2 wt%. This modification was synthesized by NAS Lab to enhance the composite's dielectric characteristics and overall material performance.

3. Experimental

3.1 Cu-Modified Ac Preparation

The process for creating copper-modified activated carbon (Cu-modified Ac) began by dissolved copper (II) acetate in distilled water at 80 °C and then allowed the solution to cool to room temperature by controlling the concentration of copper (0) of 2%wt. Activated carbon from Carbokarn Co., Ltd. was then mixed with distilled water using a magnetic stirrer. Next, the copper solution was added to this mixture, and hydrazine hydrate was carefully introduced. The mixture was reheated to 80 °C, and the resulting solution was filtered using a Buchner filter. Finally, the Cu-modified activated carbon was dried in an oven at 100 °C for 24 hours and then transferred to a desiccator to prevent moisture absorption before testing.

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3.2 Preparation of NR/ Cu-modified Ac composite

A two-roll mill (LRM-S-110/3E, Labtech Engineering Co., Ltd., Thailand) was operated at 60 °C and 60 rpm to compound natural rubber (NR) with other materials. Table 1 provides a summary of the ingredients used to form the NR/Ac-Cu composite. Mastication and compounding consisted the two steps in the composite preparation process. Initially, natural rubber was masticated in an internal mixer for roughly 10 minutes, followed by the addition of MBTs, stearic acid, ZnO, sulfur, and Ac-Cu for a further compounding phase of around half an hour. After completing the compounding process, the cure time (tc_{90}) and torque variations were assessed using a moving die rheometer (MDR). The rubber was then vulcanized through compression molding at 140 – 160 °C with an LP20-B machine, Labtech Engineering Co., Ltd.

4. Characterization

The rubber was subsequently vulcanized through compression molding at an approximate temperature range of 140 – 160 °C using a Labtech Engineering Co., Ltd. model LP20-B machine. Based on the identified cure time, NR composites were subsequently vulcanized through a compression molding process.

The hardness of the rubber composite sheet was assessed with a Shore hardness tester following ASTM D2240. Ten measurements were taken at different points, and the average hardness value was recorded.

To get to the point regarding tensile behavior—encompassing the modulus at 100% elongation, ultimate tensile strength, and elongation at break—a universal testing machine (Instron model 3365) was put through its paces. The evaluations took place at a crosshead speed of 500 mm/min, strictly in line with ASTM D412.

Table 1 Rubber compound recipe (in the unit of phr)

No	STR 5L	Stearic acid	Zinc Oxide	2,2'-Dithiobis-(benzothiazole) (MBTs)	Sulfur	Pure Ac	Cu-Modifier Ac
1	100	1	5	1	2.5	0	0
2	100	1	5	1	2.5	5	0
3	100	1	5	1	2.5	10	0
4	100	1	5	1	2.5	15	0
5	100	1	5	1	2.5	0	5
6	100	1	5	1	2.5	0	10
7	100	1	5	1	2.5	0	15

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Scanning electron microscopy (SEM) was used to investigate the cross-sectional morphology of NR/Ac-Cu composites. Specimens prepared at extremely low temperatures in liquid nitrogen were examined with a JSM-6510LV SEM from JEOL, Japan. Observations were performed at a 500× enlargement ratio under reduced pressure with an accelerating voltage of 20 kV. Prior to this, specimens underwent gold sputter deposition. A network analyzer (Agilent Technology Model E5071C ENA series) operating over the frequency range of 0.5 to 3.0 GHz was applied to evaluate the dielectric characteristics.

For electrostatic force microscopy (EFM) analysis, the rubber samples underwent a thorough cleaning process before testing. This process included ultrasonic agitation in a detergent solution (Alconox) for 30 minutes, followed by an additional 30 minutes in deionized water to remove contaminants. After cleaning, the samples were dried using a nitrogen gas stream.

Following the cleaning procedure, the surface topography and electrostatic force of the samples were analyzed using the EFM mode of a Park NX-10 atomic force microscope. The analysis was conducted in a standard room environment using a non-contact mode probe (NSC36_B), which has a spring constant (k) of 1 N/m and a resonance frequency of 90 kHz. Simultaneous topographical and electrostatic force mappings were carried out at a scanning speed of 8 μm/s, with a sample bias of 0V and a set point distance of 4 nm from the surface under ambient conditions.

5. Results and Discussion

As part of the activated carbon (Ac) modification, copper (II) from copper acetate monohydrate was chemically reduced to elemental copper. A detailed analysis was performed to assess the porosity features of both unmodified and Cu-modified Ac, with particular emphasis on specific surface area, pore diameter, and pore size. At copper loadings of 0% and 2%, a marked drop in specific surface area was observed, falling from 1195 m^2/g to 1081 m^2/g. In a similar vein, the pore volume decreased from 0.49 cm^3/g to 0.44 cm^3/g, while the pore diameter shrank from approximately 1130 μm to 1080 μm. These declines suggest the deposition of copper particulates distributed on the activated carbon surface, leading to the overall reduction in these properties.

The findings suggest that copper particle growth occurred following the reduction of the copper precursor, confirming the successful modification of the activated carbon structure. As depicted in Fig. 1, SEM images were utilized to distinguish the morphological differences between unmodified and Cu-modified activated carbon. The results further verified the deposition of copper on the surface of activated carbon particles.

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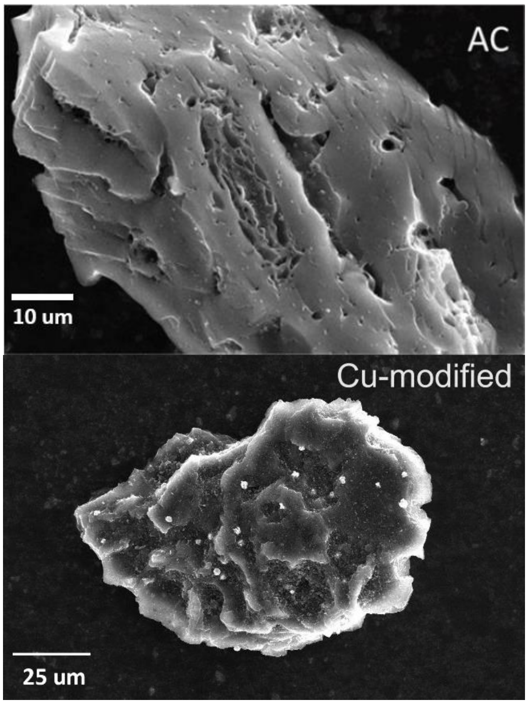

Fig. 1 SEM images of (top) pure activated carbon granule (down) Cu-modified activated carbon granule

Fig. 2 illustrates a consistent increase in density as the Ac content rises for both pure and Cu-modified samples. The Cu-modified Ac leads to slightly higher density levels at each content compared to pure Ac, suggesting that the addition of copper particles contributes to the overall mass per unit volume of the rubber composite. The density analysis showed an increase in density with the addition of Ac, as Ac has a higher density than rubber. However, the impact of Cu modification resulted in relatively minor differences in density.

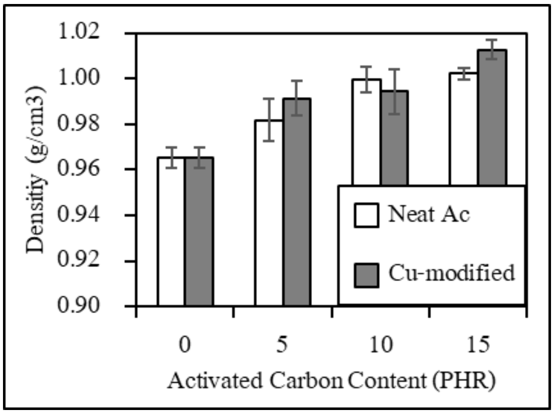

Fig. 2 The relation between Ac content and density in both pure (white) and Cu-modified Ac (grey)

Fig. 3 shows that the hardness of the rubber composite decreases as Ac content increases, with a more significant reduction in hardness for the pure Ac compared to the Cu-modified version. A decrease in hardness was observed with the addition of Ac, likely due to its interference with the rubber's crosslinking network, though Cu modification did not cause any significant changes. Notably, when the Ac content exceeded 10 phr, the expected continuous decrease in hardness was not seen, indicating that beyond 15 phr of Ac, the reduction in hardness might be less pronounced. This trend indicates that while Ac lowers the hardness, the copper modification helps maintain some of the hardness of rubber composite, likely due to differences in how the copper particles interact with the rubber matrix.

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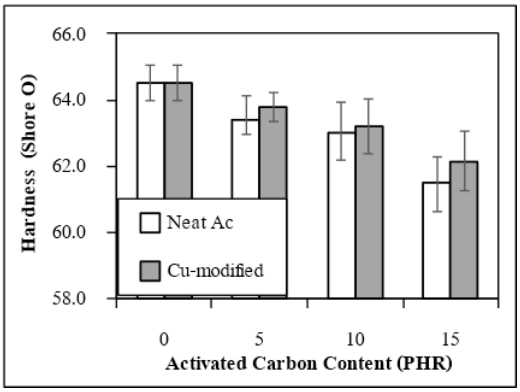

Fig. 3 Highlights correlation between Ac content and hardness in both pure (white) and Cu-modified Ac (grey)

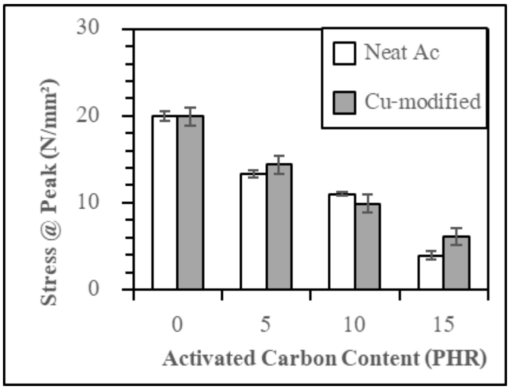

Fig. 4 Correlation Ac content and stress in both pure (white) and Cu-modified Ac (grey)

As illustrated in Fig. 4, the stress at peak results decreases with an increase in Ac content. This decline is less pronounced in the Cu-modified Ac, suggesting that the copper modification may enhance the rubber's strength and its ability to manage stress before failure. Since rubber sheets are commonly used as mats for tables or floors, it is important to establish clear testing parameters for stress and strain.

The stress trends align with the hardness results shown in Fig. 3, revealing a decrease that is inversely related to the amount of both pure and Cu-modified Ac. However, this trend does not match the strain characteristics depicted in Fig. 5, where the relationship between strain and Ac content shows only a slight reduction.

Interestingly, the rubber composite with 15 phr of Cu-modified Ac demonstrates a significant rebound, suggesting that an adequate amount of Cu-modified Ac can substantially influence the crosslinking process and improve various properties of the rubber composite sheet.

In Fig. 6, the electrical properties of the rubber composites was examined by using a network analyzer to measure the relative dielectric constant, also known as permittivity. The results show that adding Ac to the rubber compound increases its dielectric constant, which suggests an improved ability of the rubber structure to hold electrical charge. The upward trend in the graph for pure Ac may indicate a slight increase in electrical resistivity, highlighting that higher concentrations of Ac filler enhance the rubber sheet's resistivity.

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The electrostatic force measurements obtained using the atomic force microscope (AFM) indicated that rubber sheets containing Cu-modified Ac exhibited a higher average electrostatic force than those filled only with Ac, as illustrated in Fig. 7. This observation reinforces the earlier dielectric measurements, confirming that adding copper improves the rubber sheet's capacity to hold electrical charges. These findings are crucial for understanding the electrical and structural changes that take place in materials when activated carbon is incorporated during the manufacturing of natural rubber sheets.

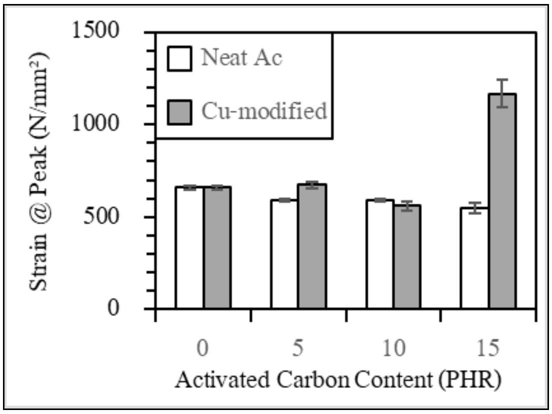

Fig. 5 Relates Ac content and strain in both pure (white) and Cu-modified Ac (grey)

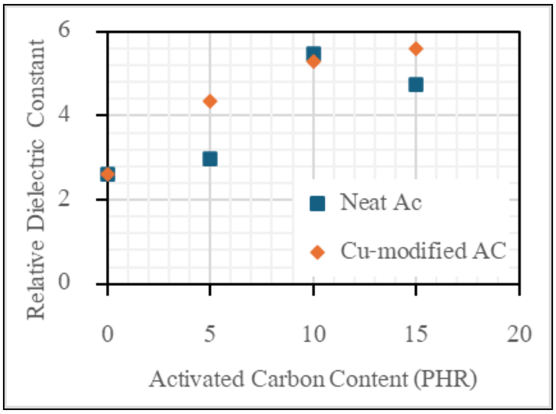

Fig. 6 Corelates between relative dielectric constant and Ac content in both pure (square) and Cu-modified Ac (diamond).

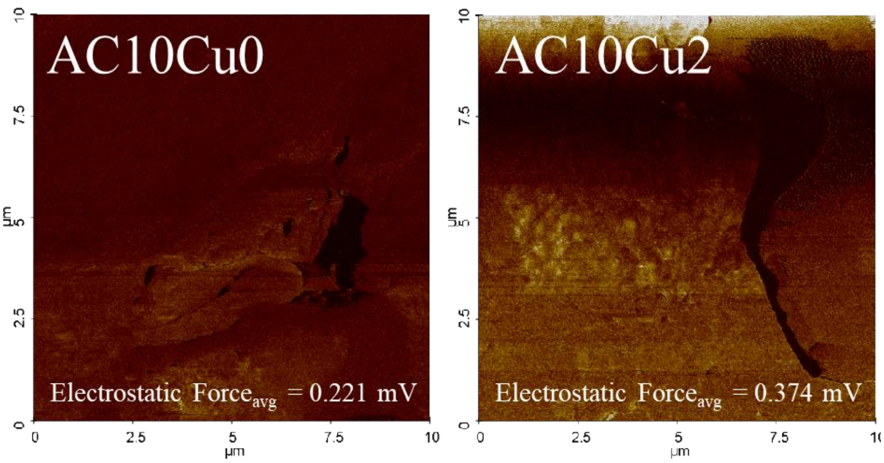

Fig. 7 Contains atomic force microscopy (AFM)

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The enhanced dielectric properties of these rubber composites have significant implications for various applications, particularly in the field of dielectric materials. For instance, the improved charge-holding capacity makes these materials suitable for use in capacitors, which are essential components in electronic devices. The ability to accumulate electrical charges efficiently can lead to better energy storage solutions, improving the performance of devices ranging from small consumer electronics to larger power systems. Furthermore, these rubber sheets can be utilized in the development of flexible sensors and actuators, where their lightweight nature and enhanced electrical properties can contribute to better functionality and durability. Additionally, the modified rubber materials may find applications in electromagnetic interference (EMI) shielding, protecting sensitive electronic components from disruptive signals. Overall, the integration of Cu-modified activated carbon in natural rubber not only enhances its electrical properties but also opens up new avenues for innovative applications in the rapidly evolving field of dielectric materials.

5. Conclusion

This study successfully demonstrates the enhancement of the electrical and mechanical properties of natural rubber sheets through the incorporation of Cu-modified activated carbon. The results indicate that the addition of activated carbon significantly influences the density, hardness, and stress-strain behavior of the rubber composites. Notably, while increasing the content of activated carbon tends to decrease the rubber's ability to handle stress, the modification with copper provides a more gradual reduction in strength, suggesting that Cu plays a beneficial role in maintaining material integrity. Electrostatic force measurements and dielectric constant evaluations reveal that Cu-modified activated carbon substantially improves the charge-holding capacity of the rubber matrix, enhancing its electrical properties. These improvements have far-reaching implications for applications in dielectric materials, including capacitors, flexible sensors, and electromagnetic interference shielding. The findings highlight the potential of utilizing Cu-modified activated carbon in natural rubber to create advanced materials that meet the growing demand for efficient energy storage solutions and electronic components.

Overall, this research contributes valuable insights into the structural and electrical alterations of rubber composites, paving the way for future innovations in material design and application in various industries especially electrostatic discharge protection materials.

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