Influence of Vulcanization System on the Mechanical Properties of CCTO/ENR50 Composite

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Influence of Vulcanization System on the Mechanical Properties of CCTO/ENR50 Composite

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Abstract. A study on vulcanization systems consist of conventional (CV), semi-efficient (SEV) and efficient (EV) was carried out by fabricating the CCTO/ENR50 composites. The rubber compound was mixed in internal mixer at 60°C and 60 rpm rotor speed for 13 min. Then, followed by hot pressing at 160°C. The effect of the vulcanization system of the composites was closely monitored on mechanical properties (stress versus strain, tensile strength, elongation at break and hardness. As a result, the CCTO/ENR50 composites with the SEV system exhibit higher modulus strength, which is related to the stiffness of the composite. Hence, it reduced the elongation at break (%) from 591 (CV system) to 495% (SEV system). It also gives high tensile strength, 5.47 MPa, and 47.74 Shore A in hardness compared to other vulcanization systems.

1. Introduction

Polymer-ceramic composite is attracting considerable critical attention to the modern electronic field nowadays. Along with the emerging of current technology, a miniature of an electronic device is essential to cope with the demand. The hybrid between these materials can overcome the limitation in each material such as brittle and high-temperature processing for ceramic material. While low dielectric constant is a classic problem in polymer material but it has a flexible characteristic that cannot be found in ceramic material [1]. However, these researches aim are to elucidate the influence of the vulcanization system on the mechanical properties of the composite. The vulcanization system composed of conventional (CV), semi-efficient (SEV) and efficient (EV) were differed by sulfur and accelerator dosage (phr). The ratio of sulfur to accelerator gives an essential influence on the final physical properties because it will affect the crosslink density of the rubber [2]. CV networks are known for their excellent mechanical strength and flex fatigue due to its network structure that consists above 65% of polysulphidic crosslinks, which make it harder to break and rearrange under stress [2]. While EV network structure has both types of crosslinks but most of the structure is monosulphidic than polysulphidic crosslinks. As a result, the mechanical strength was reduced but had more excellent oxidative aging resistance and high reversion resistance [3].

50% mol of epoxidized natural rubber (ENR50) is the modification of natural rubber using peracetic acid by converting double bonds in the *cis*-polyisoprene chain into the epoxy-functional group [4,5]. ENR50 was selected as the matrix of the composite due to their excellent properties such as thermal stability, swelling resistance and gas impermeability [6,7]. In addition, the increasing level of epoxidation also showed improvement to the rubber properties[5]. Calcium copper titanate (CCTO), is a well-known ceramic material that exhibits excellent dielectric properties. Therefore, due to the popularity owned by the CCTO as a filler for flexible polymer ceramic composites, here the several examples of the polymers material that has been studied by other researchers such as epoxy [8–10], silicone [11], polytetrafluoroethylene[1,12], polyvinylidene fluoride [13,14], polystyrene [15], polyurethane[16], polymethyl methacrylate [17] and acrylonitrile-butadiene [18].

2. Experimental

2.1. Materials

The starting materials used to synthesize $CaCu_3Ti_4O_{12}$ (CCTO) powder are calcium carbonate ($CaCO_3$), copper (II) oxide (CuO) and titanium dioxide (TiO_2) purchased from R&M Chemicals. All the chemicals used are in analytical grade. At the same time, the starting materials for fabrication of CCTO/ENR50 are epoxidized natural rubber with 50% mol epoxide (ENR-50), zinc oxide, stearic acid (activators), benzothiazyl disulfide (MBTS) (accelerator), butylated hydroxytoluene (BHT) antioxidant and sulfur (curing agent). All the chemicals used for composite compounding are in commercial grade.

2.2. Synthesis of CCTO

The CCTO powders were synthesized using a solid-state reaction technique. Firstly, raw materials were weight according to stoichiometric ratio calculation of CCTO compound then, mixed for 48 hours in polypropylene bottle with zirconia ball (the mass ratio of balls to raw materials of 10:1) at a constant speed. The wet milling technique is reliable and faster to achieve homogenous mixing; therefore, acetone was added as a grinding medium. The dried powder was calcined at 900 ℃ for 12 hours.

2.3. Fabrication and characterization of CCTO/ENR50

The CCTO/ENR50 composites were prepared by mixing process at 60℃ using an internal mixer (Brabender GmbH & Co) with 60 rpm rotor speed and capacity size of 75 cm^3. The composites were mixed according to the ingredient in Table 1 for 13 min. Then, the rubber vulcanite will leave to room temperature for 24 hours before characterization. Next, the rubber compound was pressed with compression molding at 160℃. The vulcanized sheet of rubber composite then was cut in dumbbell shape using a cutting die followed a standard from ASTM D-412 for characterizing tensile and stress-strain behaviors of CCTO/ENR50 composites. It was conducted using a universal testing machine (UTM), Tinius Olsen (model 10ST, Salfords, England) at 23±2℃ with a speed of 500 mm/min. While, for hardness properties using a standard from ASTM D2240, a 6mm thick samples were prepared for Shore A using Durometer (Instron, Massachusetts, USA). The average of ten measurements was recorded.

Table 1. List of compound ingredients and amount (phr) for three different vulcanization systems.

Ingredient/ Formula (phr)	CV	SEV	EV
ENR 50	100	100	100
Zinc Oxide	5	5	5
Stearic acid	2	2	2
BHT	1	1	1
MBTS	0.75	1.5	3
Sulphur	2.5	1.5	0.5
CCTO	0, 120	0, 120	0, 120

3. Results and Discussion

3.1. Mechanical properties of CCTO/ENR50 composite

Mechanical properties were the reaction of a material when force was applied. Their chemical structure and composition also contribute an important role to withstand the applied force. The mechanical properties of CCTO/ENR50 composites using CV, SEV and EV curing system were expressed in stress-strain behavior, tensile strength (MPa) and elongation at break (%) as illustrated in Figure 1, **Figure 2** and **Error! Reference source not found.**. Theoretically, the CV curing system has the most prominent mechanical properties because its network structure mostly consists of polysulphide (S-S bond) that can easily slide past each other to allow stress relaxation [2,3]. Surprisingly, the study showed that CV curing system only has 4.42 MPa in tensile strength, while owning the highest elongation at break (%) value,591. Despite that, the SEV system exhibit higher Young's modulus (slope liner region of the curves) than the CV and EV. It also reflects the stiffness of the composite and proved by the hardness illustrated in Figure 3. The lower mobility of the molecular chain resulted in higher stiffness and high tensile strength due to a balanced amount of crosslink bonds in the network structure[19].

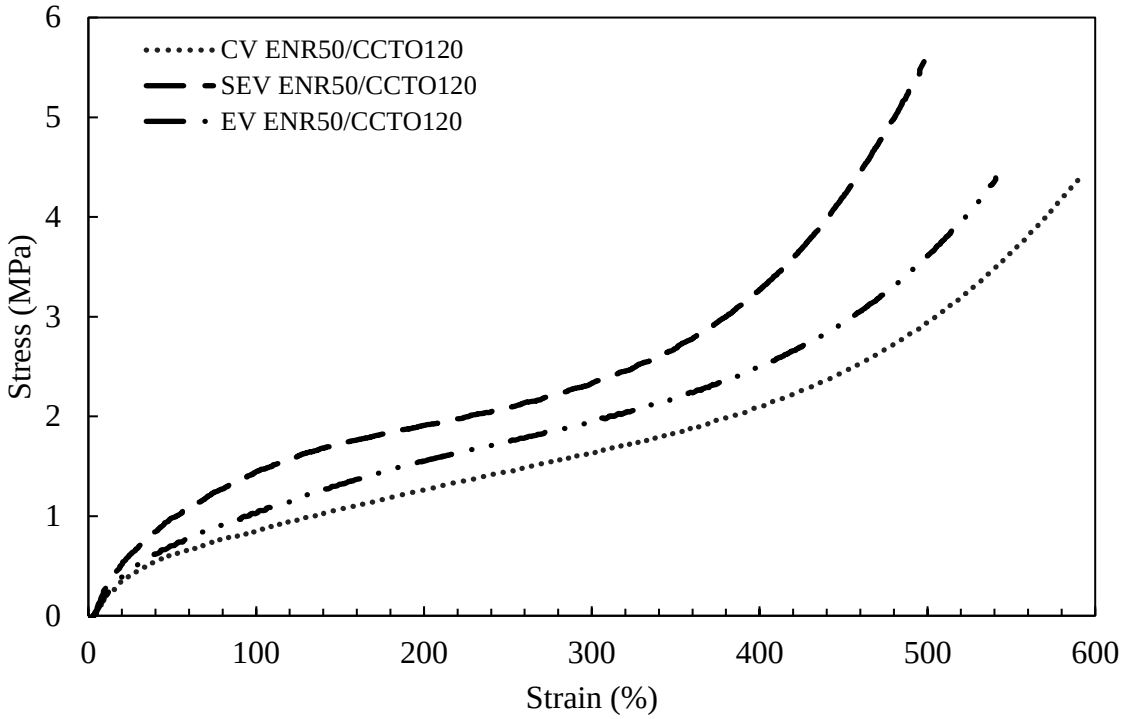

Figure 1. Stress-strain behaviors of ENR50/ $CCTO_{120}$ composites with different vulcanization systems.

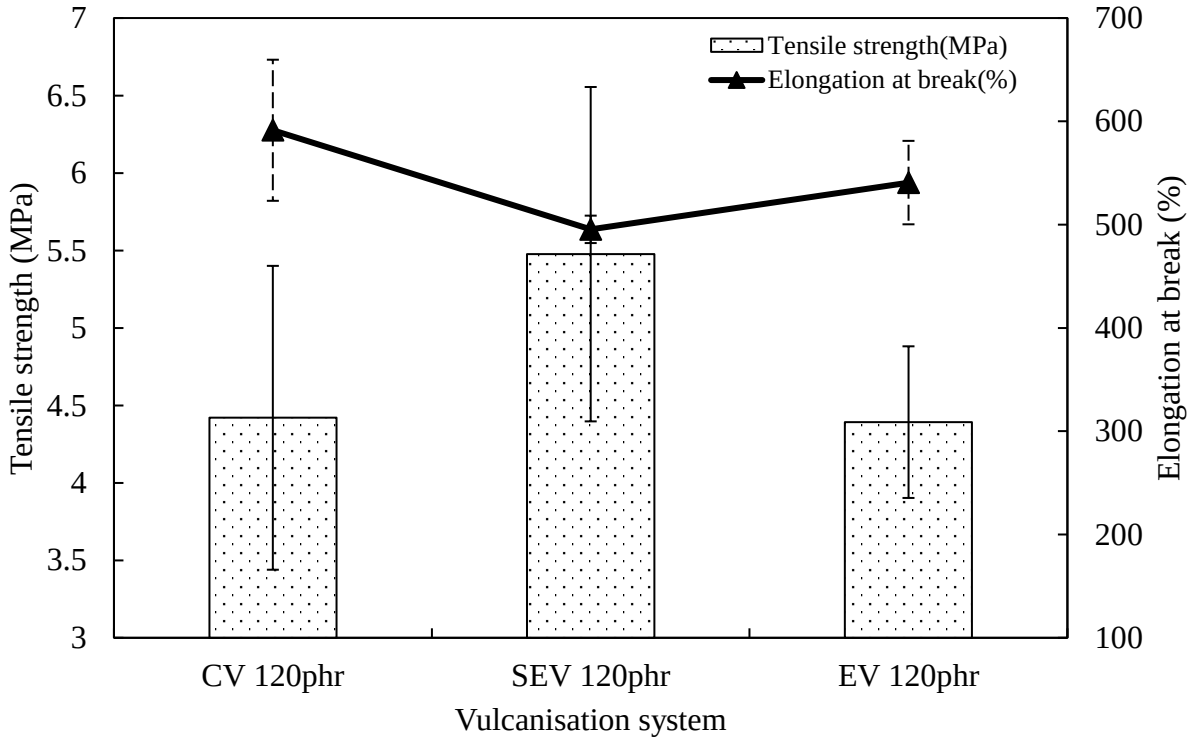

Figure 2. Tensile strength and elongation at break of ENR50/ $CCTO_{120}$ composites with different vulcanization systems.

Table 2. Tensile strength and elongation at break of ENR50/ $CCTO_{120}$ composites with different vulcanization systems.

Vulcanisation system	Tensile strength(MPa)	Elongation at break (%)
CV ENR50/$CCTO_{120}$	4.42 ± 0.981	591.36 ± 68.30
SEV ENR50/$CCTO_{120}$	5.476 ± 1.079	495.43 ± 13.276
EV ENR50/$CCTO_{120}$	4.392 ± 0.489	540.71 ± 40.388

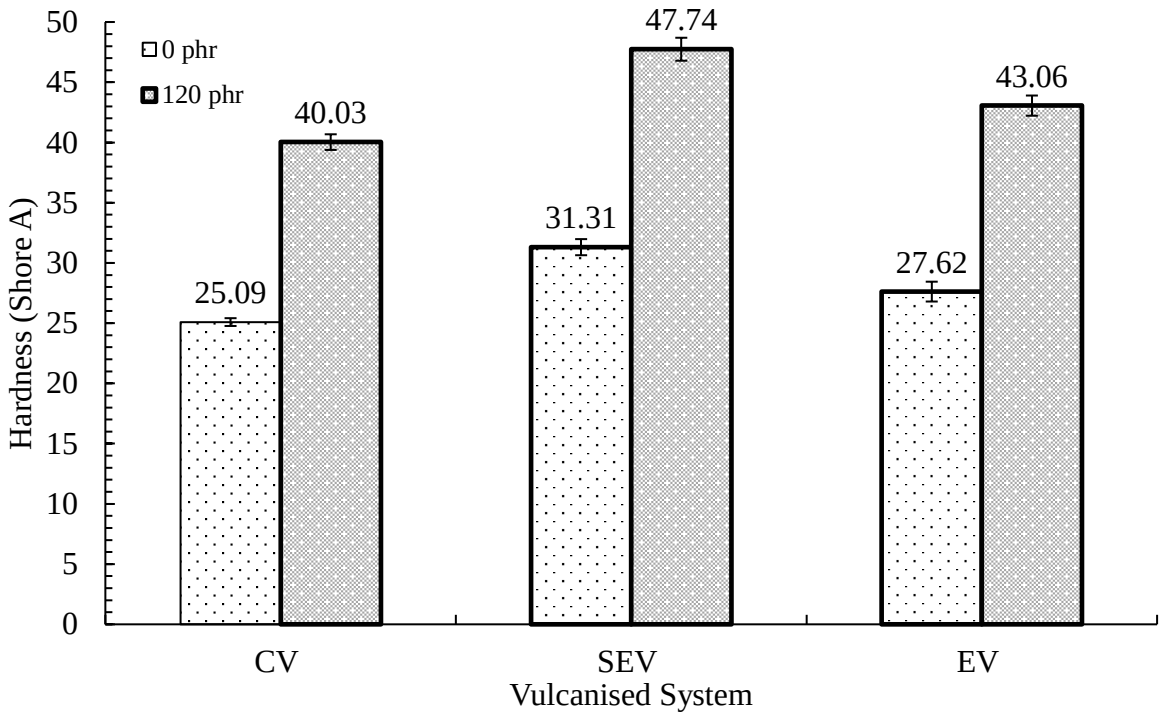

Figure 3. Hardness properties of CCTO/ENR50 composites with different vulcanization systems.

4. Conclusion

In conclusion, the vulcanization system of CV, SEV and EV has a significant influence on the rubber chain network. The vulcanization systems were differentiated by the amount of sulfur and accelerator ratio in the rubber system. Therefore, the finding showed that the composites with the SEV system have the highest tensile strength, 5.47 MPa and hardness, 47.74 Shore A.

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