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Evaluation of the properties of Perlite/EPDM Rubber

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Abstract

The effect of perlite was investigated on rheological, mechanical and flammability properties of EPDM rubbers with different perlite ratio. Perlite was added to EPDM rubber at 11.5, 24.5 and 55 phr ratios. The EPDM rubbers were compounded using a laboratory-scale Banbury mixer and subsequently vulcanized under pressure to produce standardized test plates. Rheological properties of EPDM rubber were examined by rheometer test before pressing. The tensile test, permanent deformation test, abrasion test and hardness tests were performed to determine the mechanical properties. The flammability characteristics of perlite-filled EPDM rubber were assessed using the UL-94 HB flammability test. As a result of study, minimum torque (ML) and maximum torque (MH) increased with increasing perlite content. The hardness of EPDM/20K rubber, initially measured at 58 Shore A, increased by 7.9% with the addition of 22phr perlite, reaching a value of 63 Shore A. The incorporation of 55 phr perlite into EPDM/20K rubber resulted in a 30.6% reduction in tensile strength compared to the unfilled EPDM/20K formulation. Additionally, the elongation at break values decreased (7.48%) with increasing perlite content. A similar trend was observed for compression set and abrasive wear, both of which increased (37.79% and 9.69%, respectively) as the perlite concentration was elevated. The electric current value of EPDM/20K rubber, which was 1.999mA, decreased to 0.025-0.441mA with the addition of perlite. Different ratios of perlite added to EPDM/20K rubber decreased the burning rate, while the lowest burning rate was obtained in EPDM/20K/11.5P rubber.

Keywords: EPDM, perlite, flammability, rheology, mechanical properties

1. Introduction

EPDM rubber is extensively utilized across the automotive, aviation, marine, energy, and construction industries owing to its saturated molecular structure, which imparts excellent resistance to oxygen, heat, ozone, and weathering. Additionally, its resistance to oils and hydrocarbon-based solvents, combined with high filler compatibility and oil absorption capacity, enables it to maintain its mechanical and physical properties under both static and dynamic conditions. Despite its advantageous properties, EPDM exhibits high flammability due to its low limiting oxygen index (LOI), which significantly restricts its broader application and development. Consequently, enhancing the flame retardancy of EPDM is essential [1-4].

In recent years, research efforts aimed at reducing fires caused by polymer and rubber materials have gained significant importance. Almost all polymer and rubber materials obtained from hydrocarbon sources are flammable. Therefore, the incorporation of flame retardant additives is crucial in the development of new materials to mitigate both flammability and the release of toxic gases [5–6]. Flame retardant fillers are commonly incorporated into polymer and rubber materials to inhibit, suppress, or delay the onset of combustion

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during processing. Flame retardant fillers are divided into two groups: organic and inorganic. Inorganic fillers are often preferred due to their low cost, toxicity, wear and smoke emissions. The most important inorganic flame retardants include metal hydroxides and metal hydroxyl carbonates, such as aluminum hydroxide and magnesium hydroxide, which are called "green" environmentally friendly [8-9]. The use of high filler loadings to achieve the desired flame retardant performance adversely affects the mechanical properties of the base material. Organic flame retardants usually use halogen-containing chemicals and act by scavenging reactive free radicals released by combustion in the gas phase. Although these chlorine and bromine-based flame retardants are mostly used, the use of halogen-containing flame retardants is limited or completely banned due to their environmental hazards and toxicity. [2, 3, 9]. There are different studies in the literature investigating the fireproof properties of EPDM based rubbers. Cömez and Öztürk [10] blended Ethylene Vinyl Acetate (EVM) copolymer with EPDM rubber in specific ratios to mitigate the flammability of EPDM rubber and enhance its resistance to test oils. A combination of Huntite ($Mg_3Ca(CO_3)_4$) and Hydromagnesite ($Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$) (HMCC) was incorporated into EVM/EPDM rubber blends at varying ratios. The rheological, physical, thermal, and mechanical properties were systematically evaluated of the EVM/EPDM composites. Lee et al. [11] examined the effect of aluminum trioxide (ATH) addition to ethylene vinyl acetate copolymer (EVA)/EPDM rubber blends on their thermal resistance and flammability characteristics. It was observed that EVA rubber did not affect the flame resistance of the EVA/EPDM blend. However, it was determined that the ATH significantly increased the flame resistance. In addition, as a result of the swelling test, it was determined that the swelling of the composite decreased and the solvent resistance increased depending on the ATH content. Another effective study was conducted to investigate the combustion behavior of EPDM rubber with paraffin, nanostructured hydroxide (nano-MH) and red phosphorus (RP) additions, and showed that sphere hydroxide and red phosphorus were effective in the three-dimensional network of the rubber. It has been reported that nanostructured magnesium hydroxide improves thermal stability, increases the amount of charred residue at 700 °C, and consequently improves flame resistance performance [12].

Perlite is a natural volcanic rock composed mainly of amorphous silica (SiO2, 70-76 wt%), alumina (Al2O3, 12-18 wt%) and minor amounts of other metal oxides (potassium dioxide, K2O, sodium oxide, Na2O, iron oxide, Fe2O3). Depending on the source, may also contain titanium dioxide (TiO2), calcium oxide (CaO) and magnesium oxide (MgO). Water is chemically bound in its structure, comprising between 2% and 6% by weight [13-16]. When perlite is heat treated at temperatures between 700 °C and 1100 °C, water molecules evaporate and the resulting steam can expand the perlite particles, increasing their volume to approximately 20 times their original size [17-21]. Perlite is used to protect important building elements that are not wanted to be damaged by fire due to its fire-proof properties, its ability to withstand high temperatures for a long time without deterioration, and its heat insulation properties. At fire temperatures between 700 oC-900 oC, perlite layers can protect structural steel members for up to 4 hours. [13, 19, 22-24]. With these properties, perlite has become an attractive reinforcing filler to obtain composites with lighter weight, more cost-effective, increased thermal insulation and mechanical strength, improved fire resistance properties, and to study its effects on the properties of composites. Alghadi et al [23] investigated the mechanical, thermo-mechanical and morphological behaviors of 2.5%-15% perlite filled acrylonitrile-butadiene-styrene (ABS) composites. The addition of perlite to ABS polymer led to improvements in tensile strength, elongation at break, young's modulus, and glass transition temperature and a slight decrease in impact strength and melt flow index of ABS [20]. Khongwong et al [25] investigated the effects of three different types of powder fillers such as drinking water treatment sludge (DWTS), perlite and calcium carbonate to natural rubber. As a result of the study, they stated that the tensile and elongation at break of rubber compounds gradually decreased with the increase in the amount of filler. It was determined that rubber compounds filled with small particle size filler had higher tensile strength and elongation at break than those filled with large particle size filler. Finally, it was stated that alternative fillers such as DWTS and perlite could replace calcium carbonate in the natural rubber. Özdemir [16] Investigated the effects of perlite on the combustion resistance of wood plastic composites (WPC). Perlite was used at rates of 5-10-15-20% in WPC production. According to the horizontal and vertical burning rate and LOI test results, it was reported that the burning resistance increased as the perlite amount increased. It was determined that a perlite content of 20% represented the optimal ratio for enhancing combustion resistance in wood plastic composite (WPC) production.

The aim of this study is to determine the effect of perlite on the rheological, mechanical and flammability properties of EPDM rubbers produced with different proportions of perlite addition. In addition to rheology and viscosity tests, tensile test, permanent deformation test, abrasion test and electrical strength tests were applied to EPDM/Perlite rubbers. The burning rate of EPDM/Perlite rubbers was determined by the UL-94 horizontal burning test.

2. Experimental Studies

2.1. Materials

In this study, EPDM, carbon black, white filler and other additives used in the production of EPDM rubbers are easily commercially available. Light gray colored classic perlite with a density of 750-900 kg/m3, coded Perlite Ore from Genper Expanded Perlite Ind. Trade Ltd. Co. (Kütahya, Turkey) was used as the filler material. The chemical composition of classical perlite according to the manufacturer's data is given in Table 1. In Table 2, the ratios used in the production of EPDM and EPDM/Perlite rubbers are given in phr.

Table 1. Chemical composition of perlite according to manufacturer data.

Component	Rate, %
Na_2O	3.43
MgO	0.13
Al_2O_3	12.75
SiO_2	73.00
K_2O	5.27
CaO	0.90
TiO_2	0.10
Fe_2O_3	1.00

Table 2. Formulation of EPDM/perlite rubbers

	EPDM/20K	EPDM/20K/11.5P	EPDM/20K/24.5P	EPDM/20K/55P
EPDM	100	100	100	100
Carbon Black	50	50	50	50
White filling	20	20	20	20
Oil	40	40	40	40
Zinc oxide	4	4	4	4
Stearic acid	2	2	2	2
Sulfur	1	1	1	1
MBT	0.5	0.5	0.5	0.5
TMTD	1	1	1	1
Perlite	0	11,5	24,5	55

2.2. Production and characterization

Perlite filled EDPM rubbers were turned into dough using laboratory type mini banbury. Rheometer tests were performed at 200 oC and 5 min. in accordance with ASTM D 5289 standard in Alpha MDR 2000 brand rheometer device. Test plates were obtained by vulcanizing in a press at 180 oC for 20 minutes. Tensile tests were performed in accordance with ASTM D638 standard with Zwick Z020 testing machine at 200 mm/min tensile speed. Compression set tests were performed in accordance with the DIN 53517 standard at 100 °C for

22 hours under 25% compression. Shore A Hardness tests were performed in accordance with DIN 53505 standard and at least 10 hardness measurements were taken. Horizontal flammability tests were carried out on the Fire Testing Technology-FTT-01 test device according to DIN 4102-2 (UL94/ISO 3795) standards. Conductivity test was performed in accordance with TS EN 60335-1 (DIN ISO 4649:2014-03) standard at 2000 mV and 60 s. The morphology of perlite was examined with JEOL JSM 6060 (Tekser Industrial Equipment Inc., Turkey) scanning electron microscope. Energy Dispersive Spectroscopy (EDS) analyses were also performed.

3. Experimental Results

SEM analyses were performed to examine the morphology of perlite samples (Figure 1 a). As seen in the figure, it was observed that the flake-shaped perlites were coarse, fine and unexpanded with micron size. Figure 1b shows the EDS analysis results of perlite mineral. It has been observed that perlite mineral contains Sodium (Na), Aluminum (Al), Silicon (Si), Potassium (K), Iron (Fe) and Oxygen (O) elements that form its chemical structure.

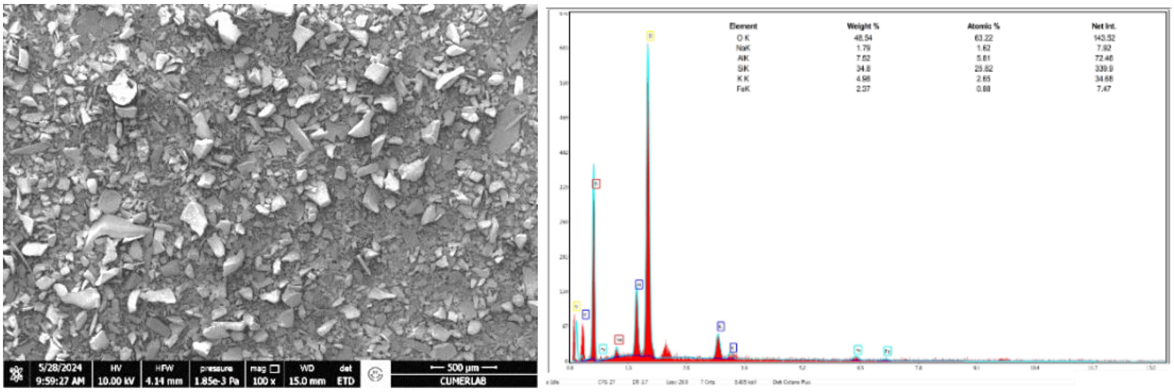

Fig. 1. a) SEM image, b) EDS analysis results of perlite mineral

Rheology test results depending on the amount of perlite are given in Figure 2 and Figure 3. As shown in Figure 2, it was observed that there was an increase in the minimum torque (ML) values of EPDM rubbers with perlite addition compared to the EPDM rubber. By adding a rijid solid phase, the maximum torque (MH) value is greatly increased. In rheology tests for EPDM rubbers with perlite addition, it was observed that the torque increase (MH-ML) increased with the addition of perlite. The increase in torque is an indication of increased crosslink density of rubbers. The hydrodynamic effect resulting from the incorporation of a hard solid phase into the elastomer can lead to increased stiffness of the materials [14]. When the initial curing time of the rubber (scorch time, ts2) and the time when the rubber reaches maximum curing (t90) are examined in Figure 3, it is observed that the ts2 time increases up to 11.5phr perlite value and decreases with subsequent perlite additions, while the t90 values increase with perlite addition. Table 3 shows the density and hardness results of EPDM and EPDM/perlite rubbers. Perlite added to EPDM rubber increased the density by 9.24%. The hardness value of EPDM rubber, which is 58 Shore A, was increased by 7.9% with the addition of 22phr perlite, reaching 63 Shore A. It was observed that the change in hardness values was related to the torque values given in Figure 2. It was related to the strong interaction between the hard particles of the perlite added as a filler material and the polymer material, resulting in an increase in brittleness and hardness values [26].

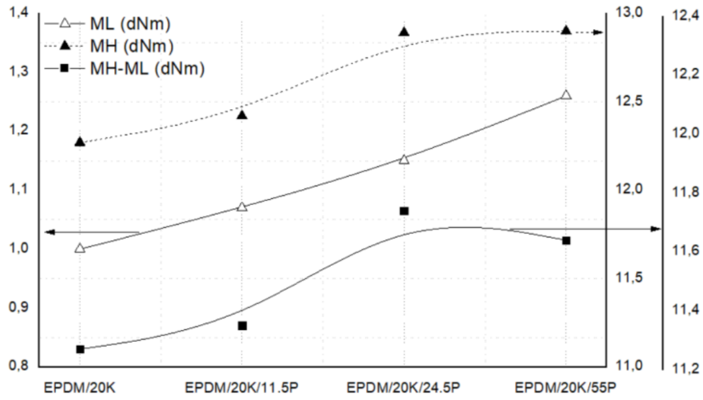

Fig. 2. Minimum and maximum torque results of EPDM/perlite rubber

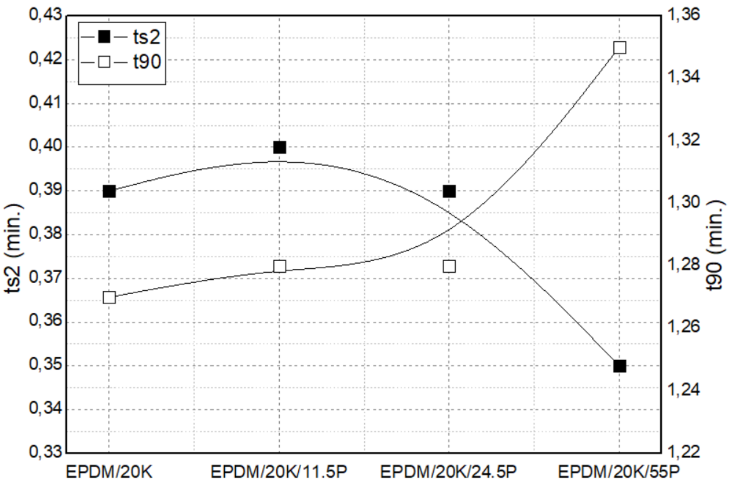

Fig. 3. T_{s2} and T_{90} results of EPDM/perlite rubber

Table 3. Physical test results of EPDM/perlite rubber

	EPDM/20K	EPDM/20K/11.5P	EPDM/20K/24.5P	EPDM/20K/55P
Density (g/cm^3)	1.08	1.10	1.13	1.19
Hardness (Shore A)	58	58	60	63

Tensile strength and elongation at break graphs of perlite added EPDM rubbers are given in Figure 4 a and b. Tensile strength and elongation at break decreased with the addition of perlite to EPDM rubber and increasing perlite ratio. The decrease in tensile strength compared to EPDM/20K rubber was obtained as 13.59% for EPDM/20K/11.5P rubber, 20.70% for EPDM/20K/24.5P rubber and 30.60% for EPDM/20K/55P rubber. The decrease in the elongation at break values of EPDM/20K/11.5P, EPDM/20K/24.5P and EPDM/20K/55P rubbers compared to EPDM/20K rubber was obtained as 2.17%, 7.73% and 7.48%, respectively.

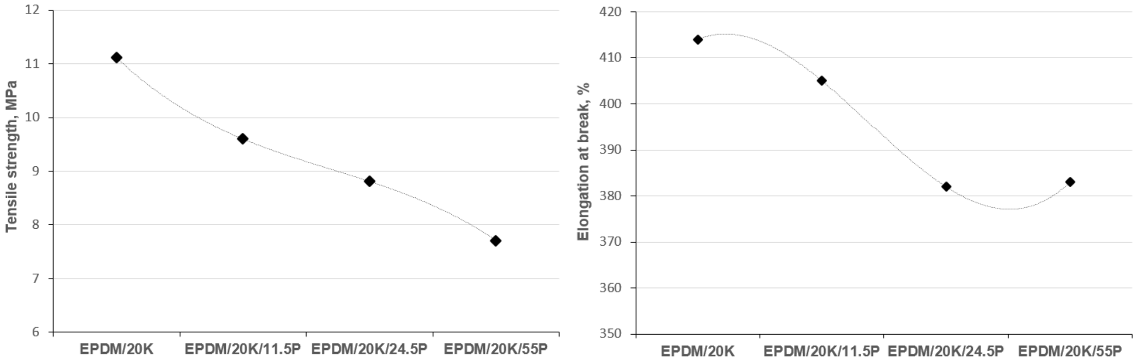

Fig. 4. a) Tensile strength, b) elongation at break results of EPDM/perlite rubber

The changes in the compression set rates and abrasive wear amounts of EPDM rubbers produced by adding different amounts of perlite are given in Figure 5 a and b. As a result of the compression set test, in which the sealing feature, which is one of the important parameters of rubber-based materials, is determined, it was observed that the compression set values increased with the addition of perlite. Compared to EPDM/20K rubber, it was determined that the compression set rate of EPDM/20K/11.5P rubber increased by 9.16%, that of EPDM/20K/24.5P rubber increased by 15.37% and that of EPDM/20K/55P rubber increased by 37.79%. When the abrasive wear results were evaluated (Figure 5-b), the lowest wear amount was obtained in EPDM/20K rubber. The amount of abrasive wear increased with the perlite ratio added to the EPDM rubber. This increase was achieved at the rates of 2.12%, 4.54% and 9.69% for EPDM/20K/11.5P, EPDM/20K/24.5P and EPDM/20K/55P rubbers, respectively.

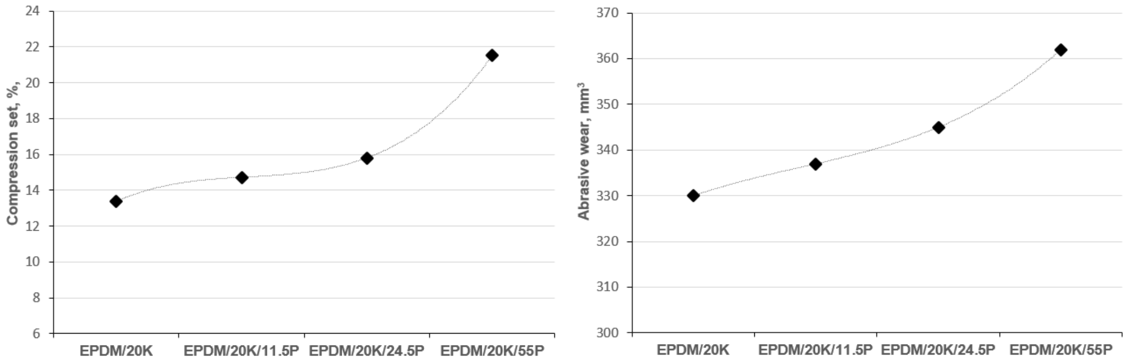

Fig. 5. a) Compression set, b) abrasive wear results of EPDM/perlite rubber

Figure 6 a and b show the electric current and UL-94-HB burning rate results for EPDM/20K, EPDM/20K/11.5P, EPDM/20K/24.5P and EPDM/20K/55P rubbers. When the electric current values in Figure 6-a are examined, the electric current value of EPDM/20K rubber, which is 1.999mA, decreased to the range of 0.025-0.441mA with the addition of perlite. Perlite added to EPDM/20K rubber at a rate of 11.5 phr reduced the electrical conductivity by 77.90%, perlite added at a rate of 24.5 phr reduced the electrical conductivity by 90.94% and perlite added at a rate of 55 phr reduced the electrical conductivity by 98.75%. In Figure 6-b, the burning rate values obtained from the UL-94-HB non-flammability test are given. While perlite added to EPDM/20K rubber at different rates reduced the burning rate, the lowest burning rate was obtained in EPDM/20K/11.5P rubber. The silica content in the perlite structure is high. While the high silica content reduces its flammability, the pores in its structure are effective in retaining flammable gases. This increases the fire resistance of perlite and the material to which it is added. Perlite also has the feature of fire resistance, remaining intact under increasing temperature conditions and preserving its thermal insulation properties.

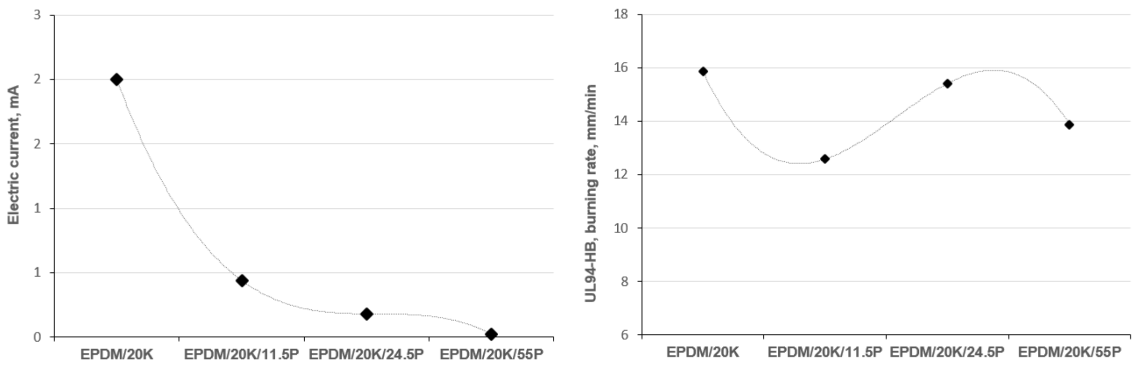

Fig. 6. a) Electric current, b) UL-94-HB burning rate results of EPDM/perlite rubber

4. Results

An experimental study of perlite filled EPDM composites reveals the following conclusions:

ML and MH values increased with the amount of perlite. The hardness value was obtained 63 Shore A with the addition of 22phr perlite by increased 7.9%.The tensile strength decreased by 30.6% for EPDM/20K/55P rubber compared with EPDM/20K rubber. The elongation at break values of EPDM/20K rubber decreased with the addition of perlite. The amount of compression set and wear increased with increasing in amount of perlite. The electric current value of EPDM/20K rubber, which was 1.999mA, decreased to the range of 0.025-0.441mA with the addition of perlite. While perlite added to EPDM/20K rubber at different rates reduced the burning rate, the lowest burning rate was obtained in EPDM/20K/11.5P rubber.

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