

## Research Article

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# Natural polymer fillers instead of dye and pigments: Pumice and scoria in PDMS fluid and elastomer composites

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**Abstract:** The electrical and thermal conductivities and light transmittance properties of silicone oil and polydimethylsiloxane (PDMS) elastomer composites were investigated. Pumice, scoria, nano-Ag, and multi-walled carbon nanotube (MWCNT) particles were used as fillers. An effective, clean, and easy method was used to prepare nanosized particles from pumice and scoria rocks. Only MWCNT–PDMS composites showed electrical conductivity. The highest electrical conductivity of  $24.7 \text{ S}\cdot\text{m}^{-1}$  was obtained with the 25% pumice–10% MWCNT–silicone oil composite. All filler particles increased the thermal conductivity of the PDMS elastomer. MWCNTs were more effective than pumice and scoria, and the thermal conductivity reached  $0.62 \text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$  with an addition of 3 wt% MWCNTs. All filler particles decreased the transparency of the PDMS elastomer. The sample with 5 wt% pumice particles reached zero transmittance. Pumice and scoria naturally colored the PDMS elastomer. The powders of these natural volcanic rocks could be used as a suitable coloring filling material instead of dyes and pigments for polymers without waste. However, it was concluded that pumice and scoria particles are not suitable for making composites with silicone oil due to the possibility of catalyzing the degradation of linear PDMS.

**Keywords:** PDMS, MWCNT, volcanic rocks, thermal and electrical conductivity, light transmittance

## 1 Introduction

Polydimethylsiloxane (PDMS) is a synthetic organosilicon polymer that has a very high market share since the 1900s and has been used in fields such as textiles, household products, pharmaceuticals, and cosmetics [1–3]. Due to their transparency, flexibility, cost-effectiveness, ease of fabrication, chemical/mechanical stability, and biocompatibility, the usage areas of polymers have increased especially in electronic devices in this century [4–7]. The experience we have gained thus far has shown us that when producing new material, we should also consider issues such as how it can be recycled when its use ends or how it affects the environment when discarded.

PDMS has different forms, such as silicone oil (in the fluid form) and silicone rubber (elastomer), in the market. The degradation of PDMS fluid has been studied by scientists. In soil, the PDMS polymer can hydrolyze to small, water-soluble siloxanols, with the ultimate product being monomeric dimethylsilanediol (DMSD). Once in the atmosphere, DMSD is expected to degrade by sunlight-induced reactions [8–11]. Lehmann et al. investigated the degradation of PDMS in a natural soil plot and, as predicted from laboratory experiments before, found that degradation is relatively rapid during the warm months of summer and slow during the cool, rainy months of fall [12]. According to Xu et al., clay minerals are catalysts for PDMS degradation, and clay type is very important in determining the degradation rates of PDMS in soil [13].

Almost every study uses PDMS in an elastomer form rather than the fluid form because of its flexibility and optical properties and because it is easier to use than silicone oil [14–16]. Although PDMS is not harmful to the environment, it cannot be expected that the elastomer form, which has been preferred in recent years, will show the same features. Because siloxanes are resistant to high temperatures, their recycling involves complex processes involving catalysts and chemicals, and more investigation into better methods for recycling siloxanes is needed [17].

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Even though PDMS is considered harmless, using different curing chemicals, catalysts, dyes, pigments, nano-sized fillers, etc., should not be considered harmless, and the hazardous effects of every component should be considered separately. For example, the natural colors of polymeric materials, which have entered every aspect of daily life, are white or transparent. However, coloring makes them more preferred. For this reason, many natural or synthetic dyes or pigments are used abundantly in production. It is undesirable for dyes to mix with the natural environment, such as soil and water. In addition to the basic properties concerning dyeing, green chemistry-related properties such as toxicity, bioaccumulation, ecotoxicity, or carcinogenicity must be considered for a proper sustainability evaluation [18].

In this study, fillers that can serve as alternatives to dyes and pigments were examined. For this purpose, volcanic rocks with their own natural and different colors were selected. Pumice and scoria are the two most abundant volcanic rocks in areas with young volcanic fields. These rocks exist in many parts of the world [19]. Pumice is an inert aluminosilicate material, and it is stable at a pH of 2.5. Pumice is biologically inert and contains no pathogens or weeds. Additionally, pumice is stable and can be reused practically and indefinitely. As a natural product, volcanic rocks can be disposed of without causing environmental pollution [20].

Then, the effects of pumice and scoria particles on the thermal properties of a PDMS elastomer were investigated. Many studies have shown that carbon nanotubes (CNTs) increase the thermal and electrical conductivities of highly insulating PDMS [21–23]. The effects of pumice and scoria added to change color and light transmittance on both forms of PDMS conductivity were also investigated in the presence of CNTs.

## 2 Materials and methods

### 2.1 Materials

Silicone oil, which is a clear, linear, nonreactive, odorless, and colorless liquid PDMS with a viscosity of approximately  $100,000 \text{ mm}^2 \cdot \text{s}^{-1}$ , was purchased from WACKER. Pumice was obtained from Bereketli Mining, and scoria from Kayseri, Türkiye. Elastosil RT 601 A/B from WACKER was used for PDMS elastomer preparation. Multi-walled CNTs (MWCNTs), with diameters of 7–10 nm and purity of 92%, were purchased from Nanografi. Silver nanopowders with sizes  $<150 \text{ nm}$  were purchased from Sigma-Aldrich and both MWCNTs and nanopowders were used as received.

### 2.2 Nanoparticle preparation

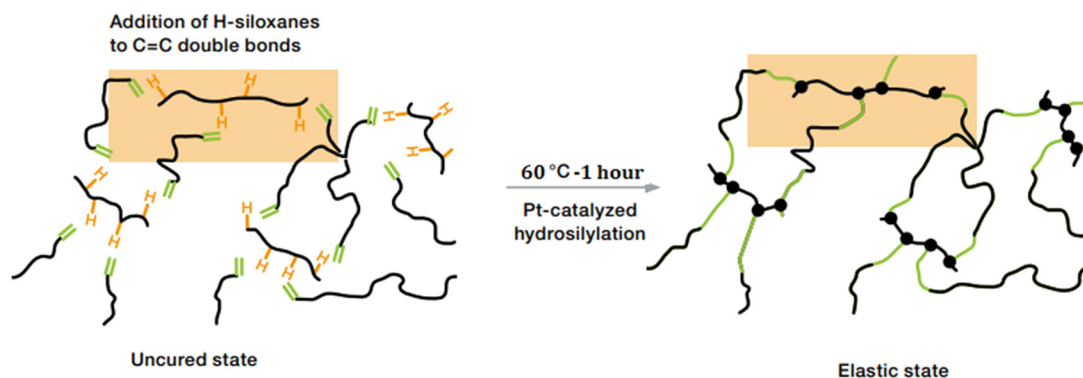
Pumice and scoria stones were ground with a Planetary Ball Mill PM 100 (Retsch) at 300 rpm for 20 min. The ground samples were mixed with 500 mL of distilled water and left for 20 h undisturbed. At the end of 20 h, 100 mL of mixture from the top was taken carefully and centrifuged at 4,000 rpm for 30 min. Particles were dried at room temperature for 2 days and used directly as fillers in the composite samples. This method is original and used in this study for the first time.

### 2.3 PDMS elastomer preparation

Component A of ELASTOSIL RT 601 contained the platinum catalyst and silicone oil, and component B was the crosslinker. The two components were thoroughly mixed at a 9:1 ratio by weight. To eliminate any air introduced during dispensing, a vacuum pump was used. The prepared mixtures were cured at  $60^\circ\text{C}$  for 1 h. Figure 1 shows the crosslink reaction of PDMS taken from the manufacturer's website (<https://www.wacker.com/h/medias/6709-EN.pdf>). During platinum-catalyzed addition curing, the crosslinker's Si–H groups react with the vinyl groups of the polymers to form a three-dimensional network. Pumice, scoria, MWCNTs, and nano-Ag were added to the mixture of A/B under vacuum conditions before heating at different weight ratios, as shown in Table 1. To prepare silicone oil mixtures, all filler particles were added simply to the silicone oil, mixed, and used directly.

### 2.4 Characterization

The particle sizes of pumice and scoria were analyzed with a Malvern Zetasizer NanoS. Particle sizes were measured in distilled water after 10 min of ultrasonication. FTIR analyses of filler particles and PDMS elastomers were performed with a Perkin Elmer Diamond 100 FTIR, and SEM-EDX analyses were performed using a ZEISS EVO LS 10 scanning electron microscope. The thermal conductivities of PDMS elastomers were analyzed using KYOTO QTM-500, and electrical conductivity analyses were performed with a KEITHLEY source meter between  $-10$  and  $+10 \text{ V}$ . Transmittance analyses of elastomer films were performed using Labsphere RSA-PE-20 attached to a Perkin Elmer UV–Vis spectrometer. Thermogravimetric analysis was performed using a Perkin Elmer Diamond TG. A total of 10 mg of the elastomer and composites were placed in a ceramic pan and heated from  $30^\circ\text{C}$  to  $1,000^\circ\text{C}$ , at  $20^\circ\text{C} \cdot \text{min}^{-1}$ , under a nitrogen atmosphere.



**Figure 1:** Crosslink reaction of PDMS in the presence of a platinum catalyst.

**Table 1:** Composite samples investigated in this study

Elastomers	Filler (%)	MWCNT (%)
PDMS/pumice	0.1	—
	1	—
	5	—
PDMS/pumice/MWCNT	1	1
	1	2
PDMS/scoria	0.1	—
	1	—
	5	—
PDMS/scoria/MWCNT	1	1
	1	2
PDMS/MWCNT	—	0.1
	—	1
	—	3
Silicone oil soft solids	Filler (%)	MWCNT (%)
Silicone oil/pumice	25	—
	50	—
Silicone oil/pumice/MWCNT	25	10
Silicone oil/pumice/nano-Ag	25	25
Silicone oil/scoria	25	—
	50	—
Silicone oil/scoria/MWCNT	25	10
Silicone oil/scoria/nano-Ag	25	25
Silicone oil/nano-Ag	—	25
Silicone oil/MWCNT	—	10

## 3 Results and discussion

### 3.1 Obtaining pumice and scoria particles at the nanoscale

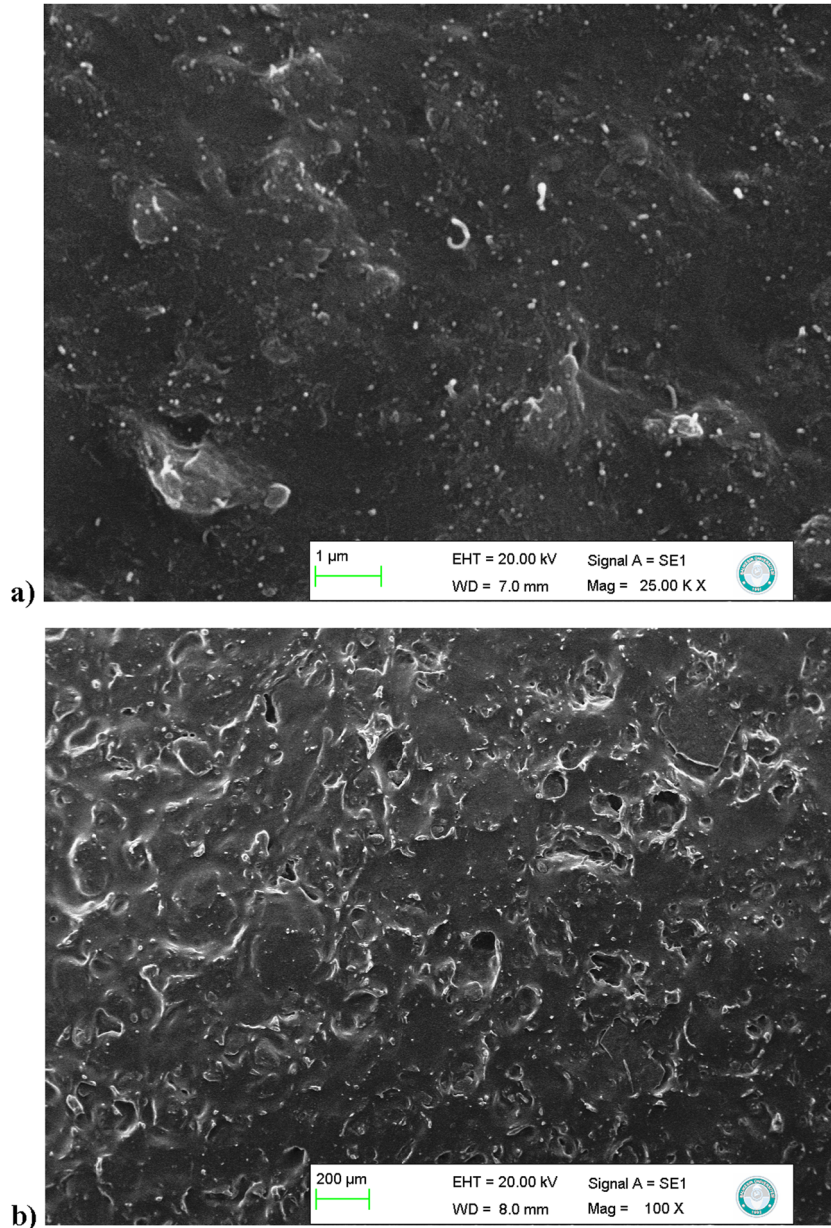
Volcanic rocks commonly come in the form of scoria and pumice. These rocks often contain high contents of silica [24]. EDX analysis shows that scoria and pumice, which are used in this study, both contain high amounts of silica

and aluminum and low amounts of Ca, Fe, Na, K, and Mg (Figures S3 and S5). Scoria and pumice have a porous structure in nature, but grinding these rocks to the nanoscale breaks up all pores, and leaf shape-like particles form from the broken layers (Figures S1, S2, and S4). By the applied method, particles  $<1 \mu\text{m}$  can easily be obtained. This method can be used for all solid particles that tend to fall to the bottom of the water according to the particle size. Approximately 657 nm of pumice and 553 nm of scoria particles are obtained from the top of the water mixture after 20 h (Figure S6). Holding in water is an effective, extremely easy, inexpensive, and environmentally friendly method for separating nanoparticles. Increasing the grinding time and holding time in water undisturbed can separate many small particles. The obtained pumice and scoria particles do not tend to stick to each other after drying and can easily disperse in PDMS while mixing.

### 3.2 Characterization of PDMS composites

All silicon oil composites numbered 1–10 are shown in Table S1 with pictures. Particles tend to aggregate at the bottom of the silicon oil and do not create a homogenous mixture. This behavior can be explained by the nonreactive and nonporous surfaces of the particles, leading to no interaction with PDMS. Conversely, MWCNTs give viscous oil a soft solid form that can be given any shape at any thickness and be easily applied to surfaces by spreading. Because of the low densities of MWCNT nanoparticles, a maximum w/w ratio of 10% is achieved with MWCNTs, while 50% is achieved with other particles.

The PDMS elastomer composites prepared in this study are listed in Tables S2 and S3, accompanied by their pictures. As seen from the SEM images, all filler particles are confined into the elastomer (Figure 2). A maximum of 5%

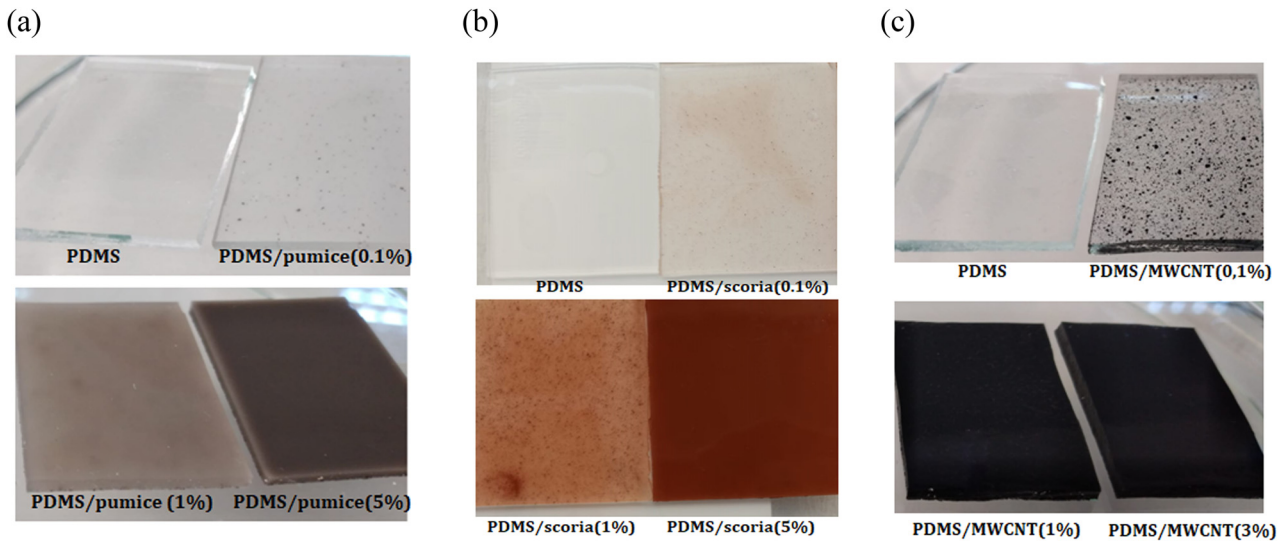


**Figure 2:** SEM images of PDMS/MWCNT/pumice (a) and silicone oil/MWCNT/pumice (b) composites.

(w/w) MWCNT particles can be packed in the PDMS elastomer, and this weight ratio cannot be used for thermal analysis because of the roughened surface of the elastomer (Figure S7). However, the specimen is suitable for electrical conductivity analysis. Figure 3 shows the PDMS elastomers with pumice, scoria, and MWCNTs in different amounts. Pumice powders dispersed in the PDMS elastomer color the transparent elastomer in different shades of gray, while scoria gives a red color. MWCNT particles added to the elastomer give a black color and make the pumice and scoria visible inside the elastomer. Synthetic dyes are known to be very toxic to the environment. Engineered

colors adversely affect all parts of life. Dyes have harmful effects on water bodies because substantial levels of industrial waste enter rivers, lakes, etc., which ultimately causes water pollution [25]. These natural volcanic rock particles can be used instead of synthetic dyes to color the polymers because they are not considered waste when discarded or mixed with the soil or water of the planet. Vu et al. developed a method for recycling PDMS that only requires a small amount of catalyst and proceeds over a wide range of temperatures (60–170°C) to efficiently yield a mixture of cyclosiloxanes (reaching 98–99% yield) from a 100 g scale of waste silicone oils [26]. Most of the filler particles or dyes





**Figure 3:** PDMS/pumice (a), PDMS/scoria (b), and PDMS/MWCNT (c) composite elastomers at different particle ratios.

and pigments present in the waste can be affected by this chemical recycling process, preventing its implementation. However, pumice and scoria can be separated from the polymer without decomposition because they are not affected by heat or solvents during polymer recycling.

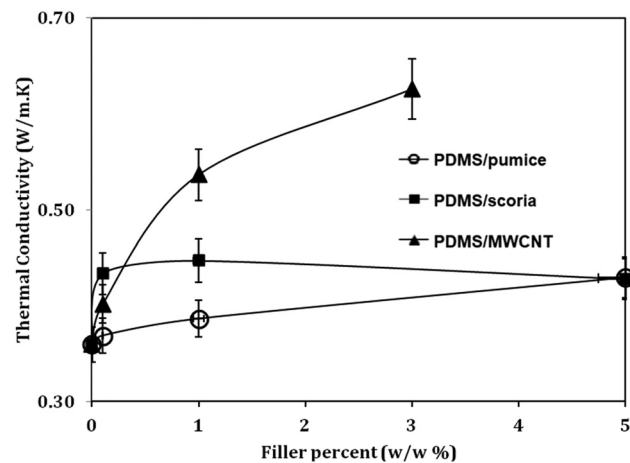
The FTIR spectra of all studied elastomers show the same result. PDMS does not chemically interact with any of the filler particles: pumice, scoria, MWCNT, and nano-Ag (Figures S8–S14). PDMS provides a surface that has a low interfacial free energy, is chemically inert, has good gas permeability and good thermal stability, and is optically transparent [27]. The inert behavior of PDMS was observed for each composite sample in this study.

### 3.2.1 Thermal conductivities of PDMS elastomers

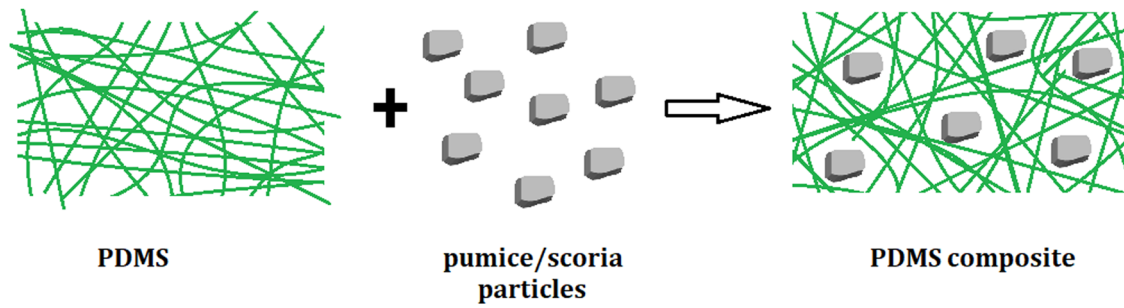
The thermal conductivity properties of polymer composites are essential for applications. The effects of pumice, scoria, and MWCNT particles on PDMS thermal conductivity were investigated in this study. PDMS has a very low thermal conductivity of approximately  $0.35 \text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$  (Table S4). Selected filler particles can change the thermal conductivity of PDMS according to the area of usage. Polymer-based dielectric materials with simultaneously high thermal conductivity and low dielectric loss are highly desirable in various applications, such as energy storage, thermal management, and electronic packaging [28]. To prevent devices from heating, filler particles such as MWCNTs have been studied by many researchers to increase thermal conductivity. Li et al. managed to increase the thermal conductivity of PDSM up to 300% by adding  $\text{Al}_2\text{O}_3$  particles [29]. Figure 4

shows that all particles increase the thermal conductivity of the PDMS elastomer. MWCNTs are more effective than pumice and scoria, reaching  $0.62 \text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$  for 3 wt%. Moreover, pumice and scoria do not greatly affect the thermal conductivity, even with increasing weight ratio, and the thermal conductivity reaches only  $0.44 \text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$ . This behavior can be useful for some fields in which insulator polymers are used, such as household products.

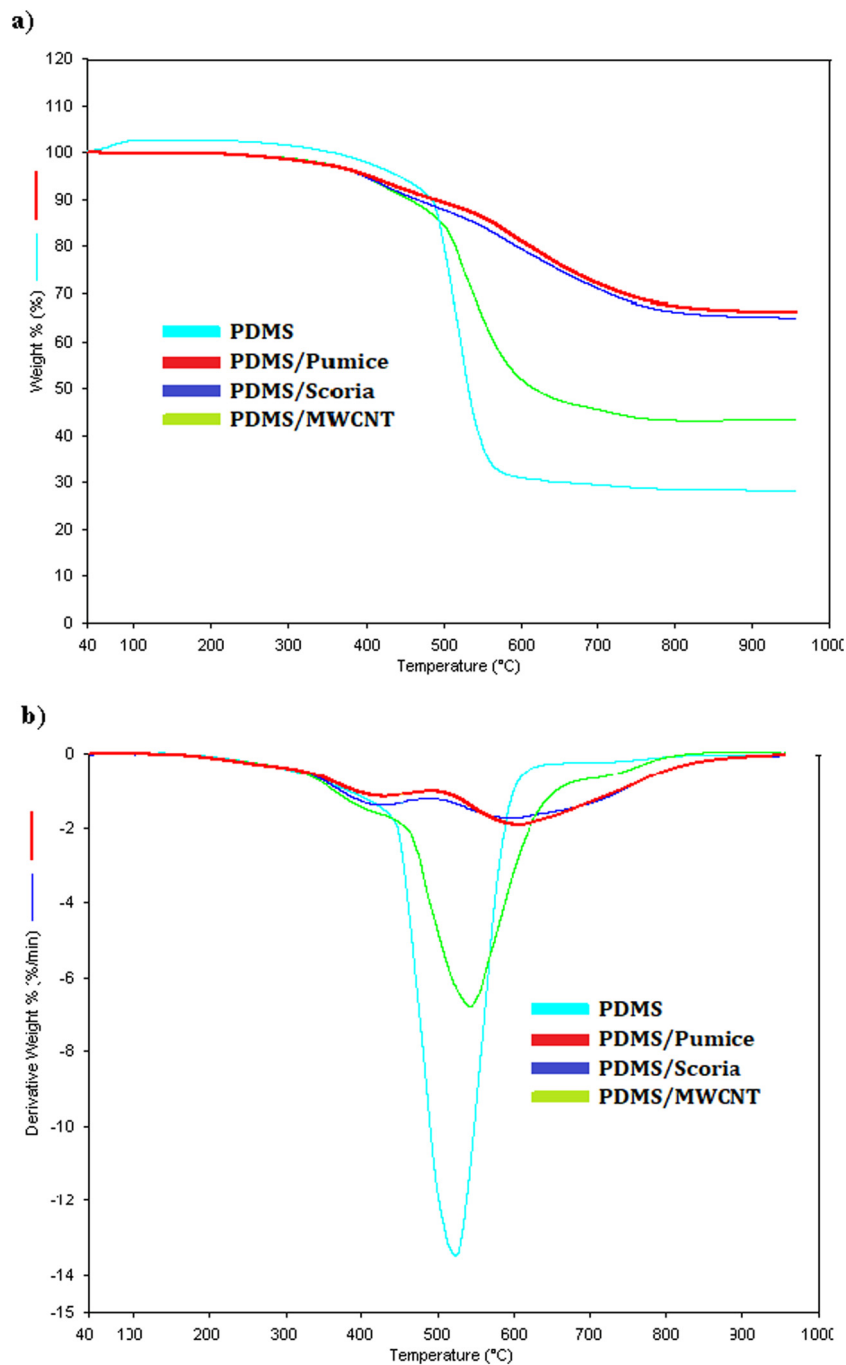
While the dense structure of the cross-links in the elastomer makes it an insulator, the fact that the filling materials create gaps in this structure may have enabled the heat to be transmitted over the particles. The possible distribution of particles in the elastomer is shown in Figure 5. The fact that pumice and scoria particles collapsed and concentrated



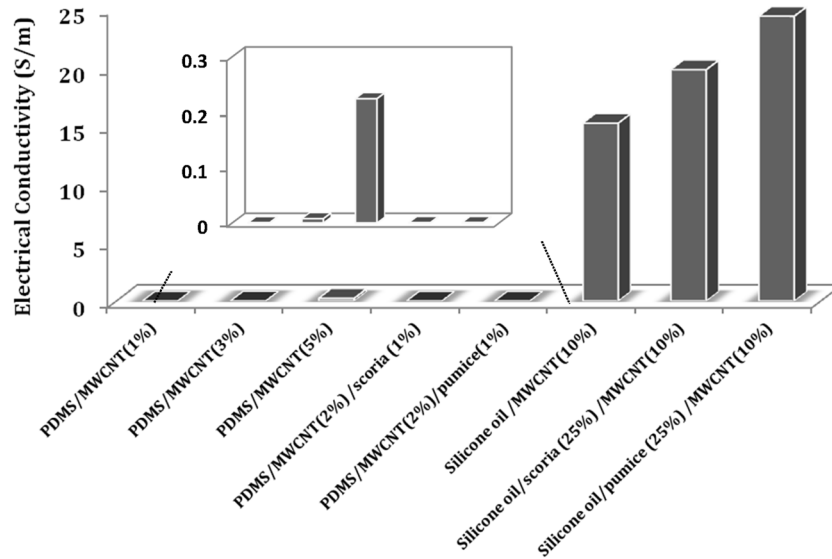
**Figure 4:** Thermal conductivities of PDMS composites with different filler ratios.



**Figure 5:** Schematic representation of the possible distribution of particles in the elastomer.



**Figure 6:** TG (a), and dTG (b) curves of PDMS elastomer and composites.



**Figure 7:** Electrical conductivities of MWCNTs including composites of PDMS and silicone oil.

at the bottom during elastomer formation enabled the elastomer to preserve its dense structure to some extent, and, as a result, the particles could affect the thermal conductivity up to a point.

### 3.2.2 TG analysis of PDMS composites

TG and dTG curves of PDMS elastomer and composites are presented in Figure 6 and data are given in Table S5. PDMS degrades at about 527°C with 77.6% weight loss, related to the exit of the lateral groups from the main chain [30]. The thermal degradation behavior of the PDMS changed in the same direction for PDMS/scoria and PDMS/pumice composites. There are two weight loss stages for PDMS/scoria and PDMS/pumice. The first step has a maximum of around 400°C with an average weight loss of 10%. The second step weight loss occurs at 580°C with around 20%. PDMS elastomer decomposes by the removal of small molecular cyclosiloxanes which occurs via the rearrangement of siloxane. Then methane is formed due to the cleavage of

the side methyl group of polysiloxane at higher temperatures [31].

While only 28% of the pure elastomer remained at 900°C, a large mass of 65% of the pumice and scoria composites of elastomer remained intact. PDMS/MWCNT composites have three decomposition steps: the first is usually associated with adsorbed apolar gases, the second is the decomposition of functional groups, and the final one, at higher temperatures, is due to the oxidation of CNTs [30]. Also, 43% residue remains at 900°C. It seems that particles in the elastomer largely prevent the formation of molecular cyclosiloxanes and the removal of free radicals from the structure.

### 3.2.3 Electrical conductivities of PDMS composites

The large aspect ratios of CNTs can enhance the electrical conductivity of the polymer composite. Electrical conductivity was studied for all composites of PDMS elastomer

**Table 2:** Comparison of PDMS composites and their electrical conductivities

Composite sample	Electrical conductivity ( $S \cdot m^{-1}$ )	Ref.
PDMS/nickel powder (10%)	$1 \times 10^{-14}$	[34]
PDMS/short carbon fiber (4%)	$1.67 \times 10^2$	[35]
PDMS/HCNT (5%)	$\sim 10^2$	[36]
MXene/cellulose nanofiber-coated PDMS foam (8%)	6.7	[37]
PDMS/MWCNT (7%)	$\sim 10^1$	[38]
PDMS/MWCNT (5%)	$2.2 \times 10^{-1}$	This study
Silicone oil/MWCNT (10%)	$1.5 \times 10^1$	This study

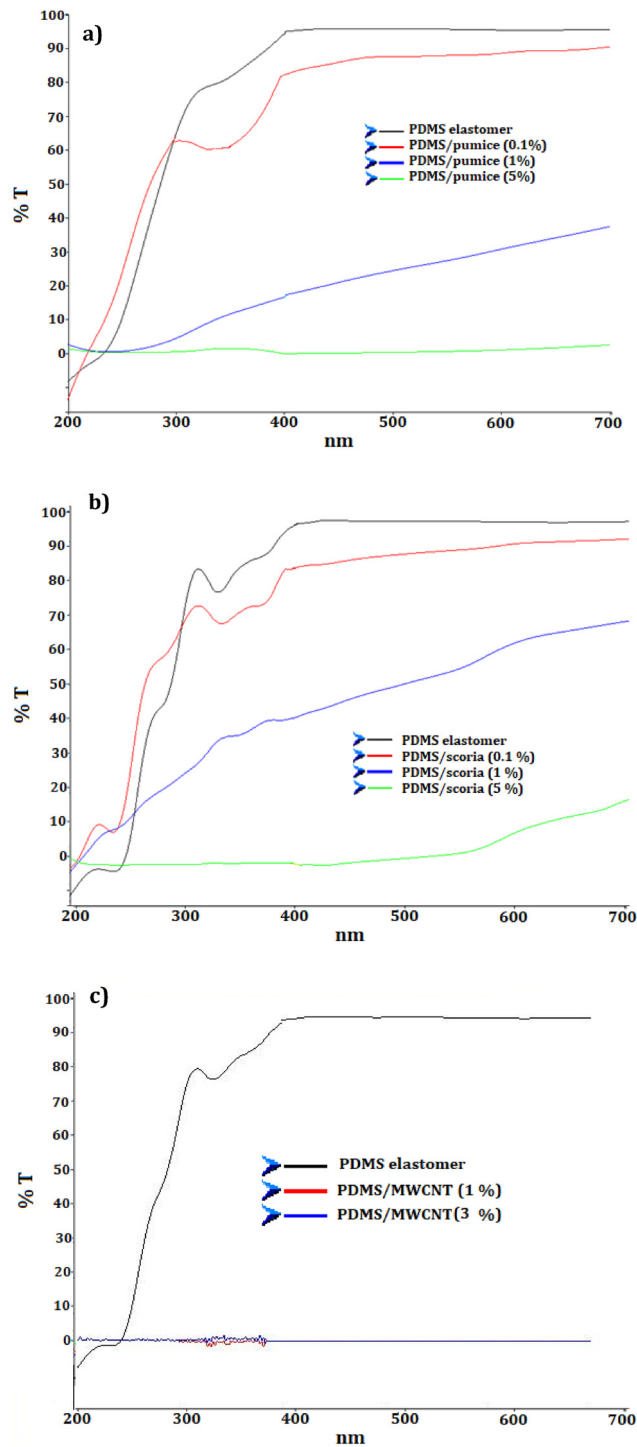
and silicon oil. Of all the composites, only the MWCNT filler shows electrical conductivity (Figure 7). Pumice and scoria particles do not change the electrical properties of either the elastomer or fluid forms of PDMS. The PDMS/MWCNT (0.1%) elastomer composite does not have conductivity because this filler ratio does not have sufficient MWCNT particles for proper contact. CNTs are regarded to be very difficult to uniformly disperse in a polymer matrix since they have very large surface areas and strong van der Waals forces, which result in aggregates [32]. The transformation duration of a viscous PDMS by curing agents to an elastomer depends on the temperature and takes some time. While waiting for elastomer formation, the added filler particles, such as CNTs, tend to aggregate at the bottom of the elastomer film. Finally, the inside of the elastomer is a heterogeneous dispersion [33]. As seen from Figure 7 and Table S6, increasing the number of MWCNTs increases the electrical conductivities of the PDMS elastomer.

Pumice and scoria particles do not contribute to the conductivities of the elastomer composites. Moreover, pumice and scoria particles slightly increase the electrical conductivities of silicone oil/MWCNT soft solids. Since the particles in the elastomer tend to collapse during curing, they cannot fill the spaces between the CNTs and exhibit a homogeneous distribution. Therefore, the particles do not affect the electrical conductivities of the PDMS/MWCNT elastomer nanocomposites. Conversely, particles settle in the spaces of the silicone oil surrounding the CNTs in soft solids, provide a homogeneous distribution, and increase the contact of the CNTs with each other; therefore, the electrical conductivity increases.

The highest electrical conductivity of  $24.7 \text{ S}\cdot\text{m}^{-1}$  is obtained with the 25% pumice–10% MWCNT–silicone oil composite. As seen from Table S6, the conductivity of the silicone oil/MWCNT composite is  $14.6 \text{ S}\cdot\text{m}^{-1}$ ; adding 25 wt % scoria increases the conductivity to  $18.5 \text{ S}\cdot\text{m}^{-1}$ . Table 2 shows some PDMS composites and their calculated electrical conductivity values. A maximum of 7% MWCNT has been doped in PDMS elastomers, while other particles can exceed this amount.

There is no electrical conductivity for the composites with Ag nanoparticles (Figure S15). The surfaces of silver nanoparticles can be oxidized, and the weight percent is not sufficient to make a continuous structure, causing the current to pass through. Similar values can be observed for PDMS/nickel powder, which is approximately  $10^{-14} \text{ S}\cdot\text{m}^{-1}$  with a 10% weight ratio [34].

The PDMS elastomer surface has a very low amount of filler particles, and all are covered with the polymer itself. When the surfaces of the elastomer composites are touched, no current flows from the electrodes, and the surface does



**Figure 8:** Light transmittance characteristics of PDMS/pumice (a), PDMS/scoria (b), and PDMS/MWCNT (c) composites.

not conduct electricity. Electrical conductivity is observed only when the electrodes are dipped into the interior of the elastomer (Figure S16). This phenomenon makes the composite an insulator from the outside and a conductor from the inside. This result is not the case with silicone oil composites.



### 3.2.4 Light transmittance of PDMS composites

PDMS is an ideal elastomeric material suitable for optical and electronic devices due to its flexibility, high optical transparency, nontoxicity, and low cost [39]. Figure 8 shows the light transmittance characteristics of PDMS/pumice, PDMS/scoria, and PDMS/MWCNT elastomers. As seen from the figure, the PDMS elastomer transmits almost 100% of visible light but the transmittance decreases sharply between 400 and 200 nm, eventually decreasing to zero. MWCNT particles starting from 1 wt% block the light transition of elastomer composites. The addition of 0.1% pumice and scoria particles decreases the elastomer transmittance by 10% in the visible region. The sample with 1 wt% pumice decreases the transmittance to 40% and with scoria decreases to approximately 60% in the visible region, with both decreasing to zero in the UV region. The sample with 5 wt% scoria/PDMS still exhibits transmittance at 20%, while that of the pumice/PDMS elastomer decreases to zero. PDMS remains semitransparent at a low filler particle content.

Solar cells and electronic devices are easily affected by external humidity, light, heat, and chemicals. There are a wide variety of studies available to protect them from the effects of the external environment. For example, Wei et al. obtained a safe and environmentally friendly encapsulation material that avoids the structural damage of perovskite solar cells under high-temperature or UV-curing conditions [40]. Naturally colored PDMS elastomers with pumice and scoria can be preferred as environmentally friendly materials for the encapsulation of electronic devices or solar cells. These volcanic rock particles are not damaged by external factors, such as dyes and pigments. There is no expiration date or decomposition time.

## 4 Conclusions

In this work, the fluid forms of PDMS (silicone oil) composites with PDMS elastomer composites were compared. The electrical and thermal conductivities and light transmittance properties of the composites were presented. An effective, clean, and easy method was used to prepare nanosized particles from pumice and scoria rocks. MWCNT particles made the PDMS elastomer electrically conductive, but the conductivity was limited by achieving the maximum filler amount. However, silicone oil was mixed with double the amount of MWCNTs, which led to a sharp increase in electrical conductivity. Additionally, pumice and scoria particles supported the structures of silicone oil/MWCNTs, and the conductivity increased again.

Silicone oil/MWCNT composites formed a soft solid structure; thus, these composites could be easily applied to any desired surface at any thickness or extent. A conductive surface could be applied and discarded from the surface easily while recycling. Pumice, scoria, and nano-Ag particles did not affect the electrical conductivity of PDMS. The thermal conductivity of the PDMS elastomer only improved with MWCNT particles, and mixing pumice and scoria particles with PDMS did not significantly change the thermal insulator features. All filler particles decreased the transparency of the PDMS elastomer. The addition of 5 wt% elastomers reached zero transmittance with pumice particles.

Silicone oil decomposed naturally in the soil environment. However, the pumice and scoria particles improved some properties of silicone oil composites, and their effects on the decomposition of silicone oil should be investigated because they are natural volcanic rocks abundant in soil. Finally, the environmental effects of composites were very low because silicone oil is harmless when discarded to nature; since pumice and scoria are natural rocks, they were not considered waste. MWCNTs could be taken inside the silicone oil composite and reused after dissolving the polymer with a solvent. Pumice and scoria particles are inert; thus, PDMS elastomer recycling with chemicals or heat would not be affected by fillers.

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**Author contributions:** Pınar Turan Beyli: writing – original draft, methodology, and formal analysis. Esra Özvezir: experimental and analysis.

**Conflict of interest:** The authors state no conflict of interest.

**Data availability statement:** The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request. The supplementary materials section can be viewed on the GREENPS website.

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