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Experimental Study of the Effect of Tetraethoxysilane on Mechanical Properties of Silicone Rubber - Polyurethane Blend

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Abstract

Mixing polymeric materials to obtain a blend is considered very important. To improve mechanical properties, as the extent of compatibility and the type of bonding between the blend components greatly affect the properties of the resulting blend, especially the mechanical properties and physical properties in general. The current research shows that it is important to study how compatibility and chemical and physical bonding affect the mechanical and other properties of the polymeric blend made when thermosetting is used. Herein, silicone rubber (SR) is mixed with polyurethane (PU) with the help of a substance called tetraethoxysilane (TEOS) to improve the compatibility and chemical bonding between the components of the blend. This was done by studying the internal structure of each material separately and the possibility of compatibility between the blend's components. Besides, mixing (80%) resin of SR with 0.1% (TEOS) and then mixing with (20%) polyurethane and then adding hardeners for each one, respectively, to form an interpenetrating polymer network. The findings show that a mixture (IPN) is physically and chemically bonded, and this indicates an increase in some mechanical properties as the tensile strength increased to 0.5 KPa. Tear strength increased to 12 KPa, which also increased the number of fatigue cycles to (3,100,023 cycles, and a decrease in the swelling ratio was also observed to 3.4 %. The examination of Fourier transforms infrared spectroscopy (FTIR) confirmed the existence of chemical bonds (crosslink) between (SR) and (PU) through (TEOS). Scanning Electron Microscopy (SEM) also confirmed the decrease in micro-gaps, which are the starting point for failure in most mechanical properties.

Keywords: *Silicone rubber; Polyurethane; Tetraethoxysilane; Mechanical properties; Grafting agent.*

Introduction

Recently, scientists and engineers in medical sciences have tended to study and conduct research about finding medical alternatives that are close to the human body and the missing organs of the body, and this finds an opportunity to compensate for the missing parts and reduce human suffering [1]. In the last forty years, the trend has turned to biomaterials and polymers compatible with the human body that are non-toxic and have no collateral damage [2].

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A new blend with better specifications than each material separately in terms of physical, chemical, and mechanical properties. Vuillequez et al[3].studied the effect of tetraethoxysilane (TEOS) on silicone rubber in terms of mechanical properties and how to increase them.

Huang et al[4]. studied the formation of silicone and polyurethane coatings, where the material is heat- and corrosion-resistant. In details, the study discussed the successful synthesis of octa(dimethylethoxysiloxy) POSS (ODES) and its application as a novel curing agent for preparing room-temperature vulcanization (RTV) silicone rubber (SROD). The authors claimed that SROD using ODES as a cross-linker demonstrates significantly better mechanical properties compared to SRTE and reported that the highest tensile strength of SROD reaches 1.26 MPa, which is three times that of SRTE. Mikhailova et al[5].discovered silicone bonding with polyurethane chemically, according to the US patent registered with the number (US 8,354.478 B2). worked on mixing silicone rubber with polyurethane-protected material (as an emollient or water-repellent material with favourable film-forming characteristics). He used weight ratios for silicone and polyurethane ranging from 0.5% to 99.5%. In addition, by mixing these two materials in a ratio of 1:1, a soft paste with high viscosity and lustre was obtained. The product was used in cosmetics, hair shampoo, toothpaste, shaving paste, antiperspirants, skincare, and soap.

Panmanee et al[6].studied the preparation of a cross-linked blend between silicone rubber and polyurethane rubber in the presence of a grafting material (silane group), which creates crosslinking between the chains and thus increases the physical properties as well as thermal stability. Maity et al[7].used modified silicone rubber with polyurethane to make a coating for high-voltage electrical wires. Where he used 70 g of polydimethylsiloxane, 30g of SI-PU, 90 g of CaCO3, and 7 g of methylethylketone. And it took 19.1 min for drying and tensile strength (3.2 Mpa) and thus obtained a breakdown stress greater than 20 KV and a flame resistance at the level of FV-1.

Wang et al[8]. found that silicon is the most compatible material with almost all polymers. Its mechanical properties are close to those of the human body: compatibility, sterilisation ability, and chemical inertness against liquids and the body. In their study, Li et al[9]. investigated the effects of various graft polymers as compatibilizers on a 70/30 blend of natural rubber (NR) and silicone rubber (SR). The researchers observed enhancements in compatibility and mechanical properties, particularly an increase in tensile strength when the graft ratios were elevated. The utilization of reactive and non-reactive compatibilizers resulted in disparate consequences. In addition, Qian et al[10]. reported that limonene, from orange peel or microbial engineering, broadens applications in silicone rubber through LTES synthesis, enhancing strength and heat resistance. In their study, Moolsin [11]. investigated the effects of various graft polymers as compatibilizers on a 70/30 blend of natural rubber (NR) and silicone rubber (SR). The researcher observed enhancements in compatibility and mechanical properties, particularly an increase in tensile strength when the graft ratios were elevated. The utilisation of reactive and non-reactive compatibilizers resulted in disparate consequences. According to the research conducted by Feng et al[12]. their study involved the synthesis of high-molecular-weight silicone rubbers denoted as P2 and P3. These rubbers were prepared through the combination of certain siloxane compounds, followed by crosslinking utilising a distinctive technique. The resulting silicone rubbers exhibited an improved modulus when compared to the conventional silicone rubber variant, P1. The confirmation of this mechanism was achieved by the utilisation of solid-state nuclear magnetic resonance (NMR) and spectroscopic techniques.

Based on the above survey, in the field of cosmetics, there is a pursuit to develop polymers that closely mimic the properties of natural polymers found in the human body. Achieving this goal necessitates thorough research into the intricate relationships between various materials and the discovery of novel substances possessing similar attributes.

Additionally, innovative approaches such as the creation of interpenetrating polymer networks (IPNs), which involve the interplay of two or more materials, are being explored. These endeavors rely heavily on rigorous experimentation and comprehensive studies. Therefore, the present study aims to improve the bonding and crosslinking between silicone and polyurethane to increase the mechanical properties of the blend by grafting with the silane group.

Experimental design

Materials under consideration

In the current work, imported rubber-silicone from the Company of Technology was utilized (Sunny Zhou), RT-800 silicon rubber, a platinum hardener used with silicon. Also, Polyurethane (PU) is supplied by Henkel Poly Bit Industries company, (UAE). Polyfoam I-20 is a two-component, polyol the resin and MDI (isocyanate) the hardener. Tetraethoxysilane (TEOS) is a group of silanes from the company (Glentham Life Sciences) from the United Kingdom, with a molecular weight of (208.33) and a chemical composition ($C_8H_{20}O_4Si$) and it is a transparent substance without colour, as well as the purity of the product, which is (99%).

Preparation IPN Blend

Preparation of SR/Pu IPN Blend

After obtaining the best ratio from several sources (5, 8), which is the ratio (SR 80% + Pu 20%), the resin of the silicone was mixed with the resin isocyanate of the polyurethane to make a blend (IPN) in a mixer at a speed of 45 rpm for 10 minutes, then the polyol (hardener) of the polyurethane was added and mixed for three minutes until the mixture became one phase in terms of appearance, then it was added to the hardener of the silicone and mixed for two minutes. Then it is placed in the mould, placed in the pressure device, and subjected to a pressure of 60 psi for 24 hours. Then it is taken out and placed in an oven to dry at a temperature of 120 °C for 8 hours, and then it is ready to conduct laboratory tests, as Fig. (1-a).

Preparation of SR-Grafted/Pu IPN Blend

The same previous method of preparation is repeated, but (0.1%) from (TEOS) is added to the silicone resin and mixed for two minutes, and then the previous steps are completed, as shown in Fig. (1-b).

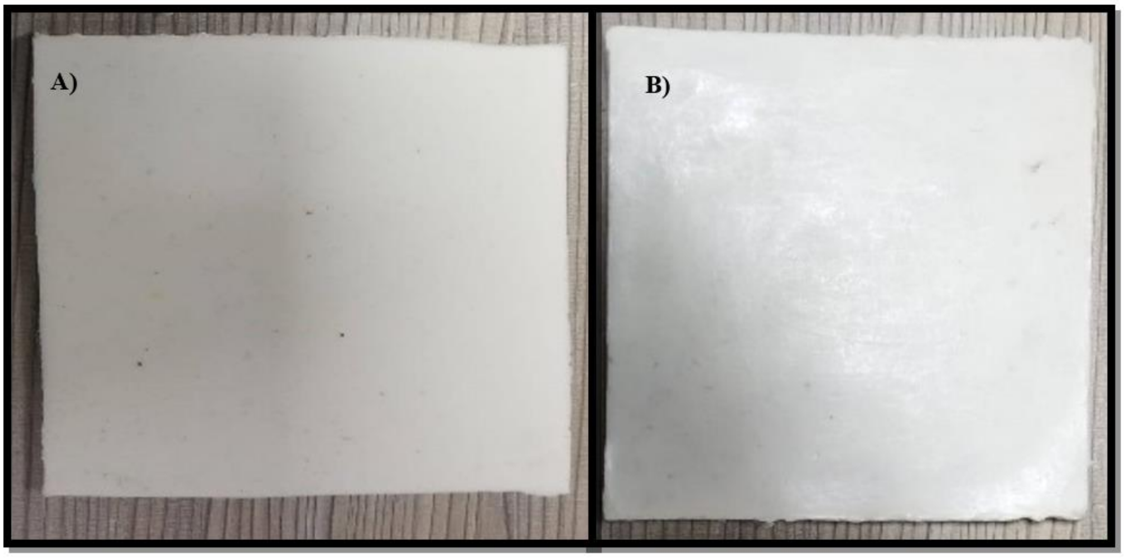

Fig. 1: Prepared samples: (A) (SR 80% + Pu 20%), and (B) (SR 80% + Pu 20% + 0.1%TEOS)

Applied tests

Tensile Strength

Preparation of tensile samples by the international standard has been adopted with ASTM-D412 as shown in Fig. 2, to prepare the samples with the required dimensions and perform the tensile test[13]. The test sample is in Fig. 3.

Fig. 2: Test Samples of Tensile testing (A, B-before test, C, D -after test)

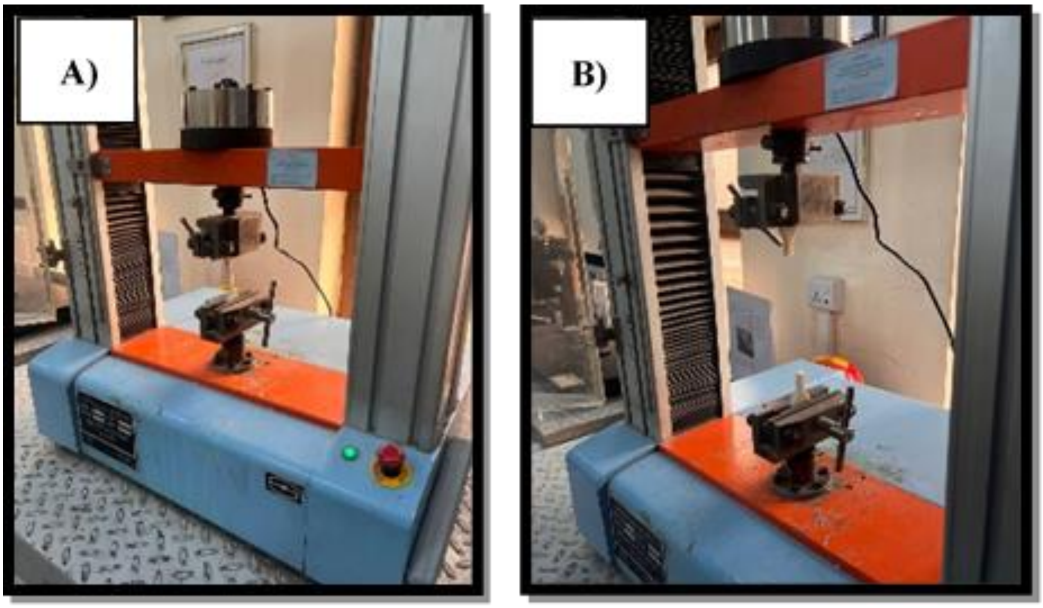

Fig. 3: Tensile Strength Device (A -Before test, B-After test)

Tear test

On the same universal testing machine in tension, the tear strength in rubber is shown in Fig. 4. A measure of a material’s resistance and preparation sample to tearing force is determined per ASTM D624. The examination was The General Company for Rubber Industries and Tires/Babylon, as shown in Fig.5.

Fig. 4: Tear Device

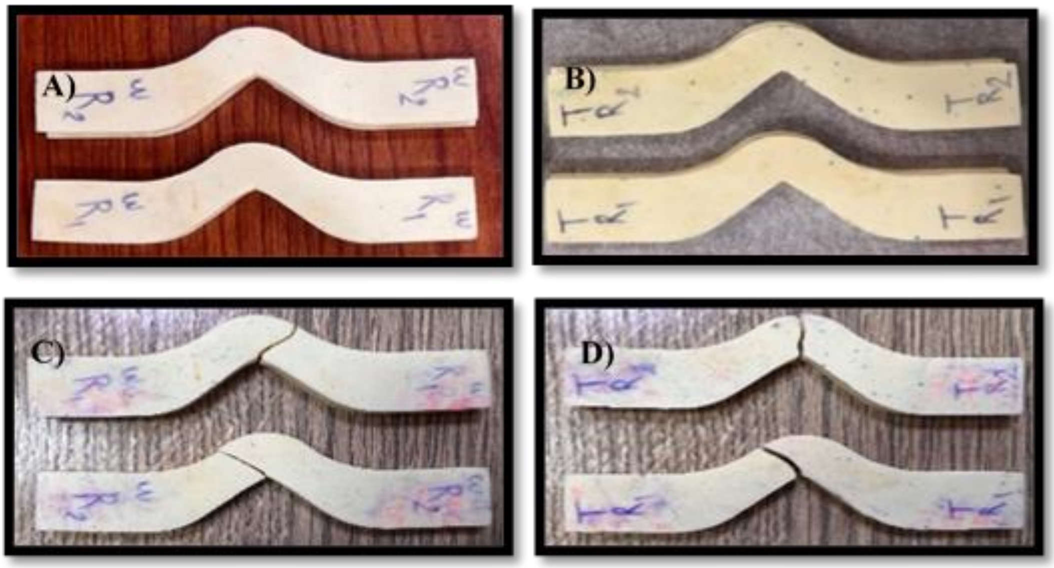

Fig. 5: Test Samples of Tear Testing (A, B-before test, C, D-after test)

Compression test

The international standard specification (ASTM D395) has been adopted to prepare the samples at the required thickness of 12.5 mm and to carry out, as shown in Fig. 6, a load-shedding test of 120 kg for 6 hours and after removing the applied load for 30 minutes to determine the extent of the compressive strength and return to the original shape of the samples prepared in the above proportions, as shown in Fig. 7.

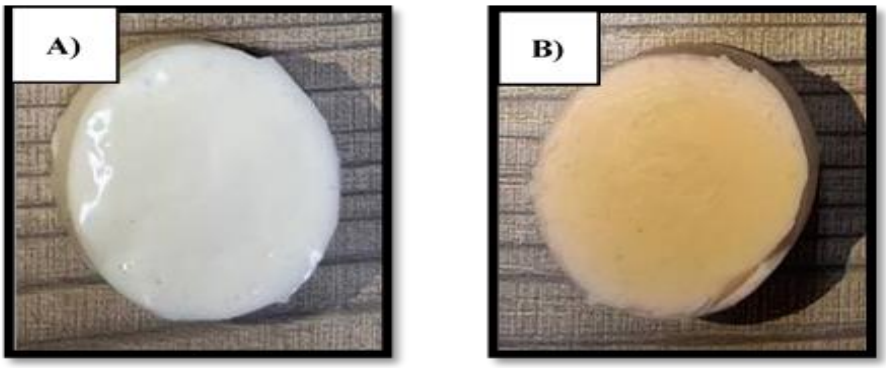

Fig. 6: Test Samples of compression: (A) (SR+ Pu), and (B) (SR+ Pu+ TEOS)

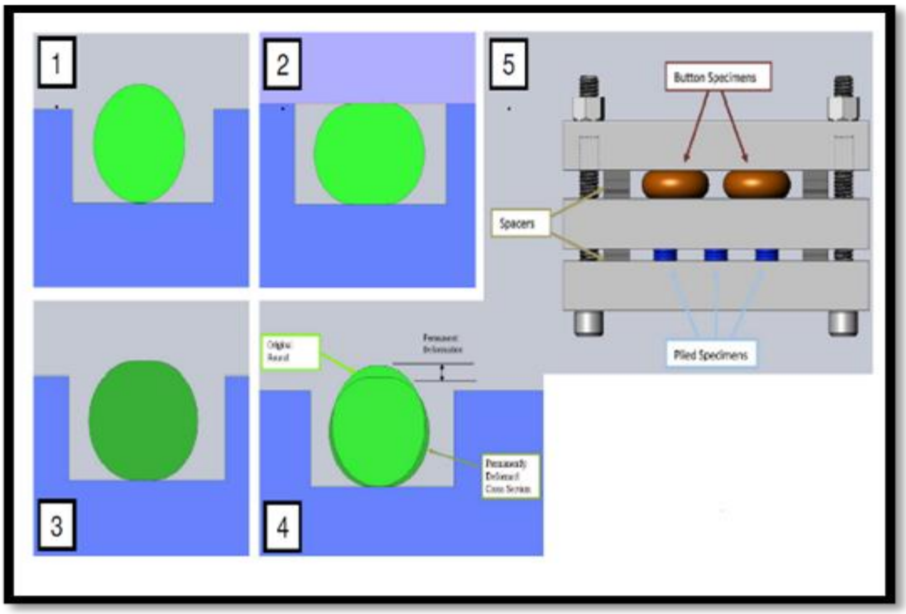

Fig. 7: Compression test[11]

Hardness

The hardness test is performed according to ASTM D2240 by the Durometer hardness test, type (Shore A). The surface of specimens must be smooth in zone testing, as well as test sample preparation according to specification dimensions.[14]. as shown in Fig. 8.

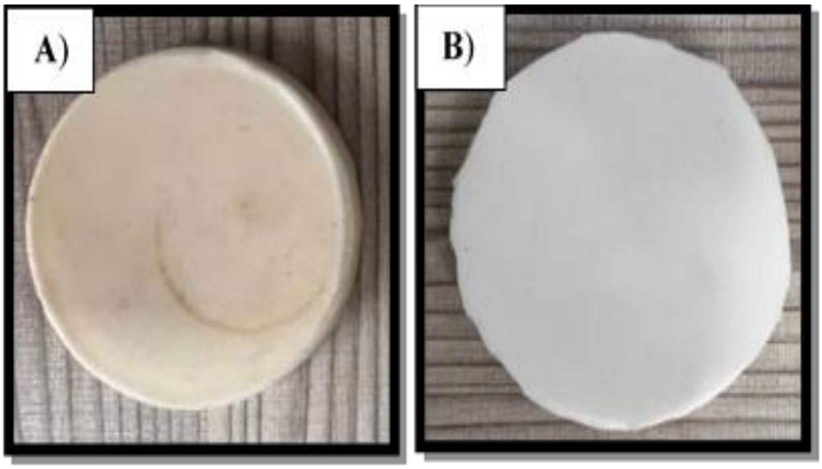

Fig. 8: Test Samples of Hardness: (A) (SR + Pu), and (B) (SR+ Pu+ TEOS)

Fatigue test

The sample was prepared according to the standard specification ASTM D430, with two samples for each previously mentioned type, as shown in Fig. 9. and the examination was carried out on the fatigue device,[15]. as shown in Fig. 10.

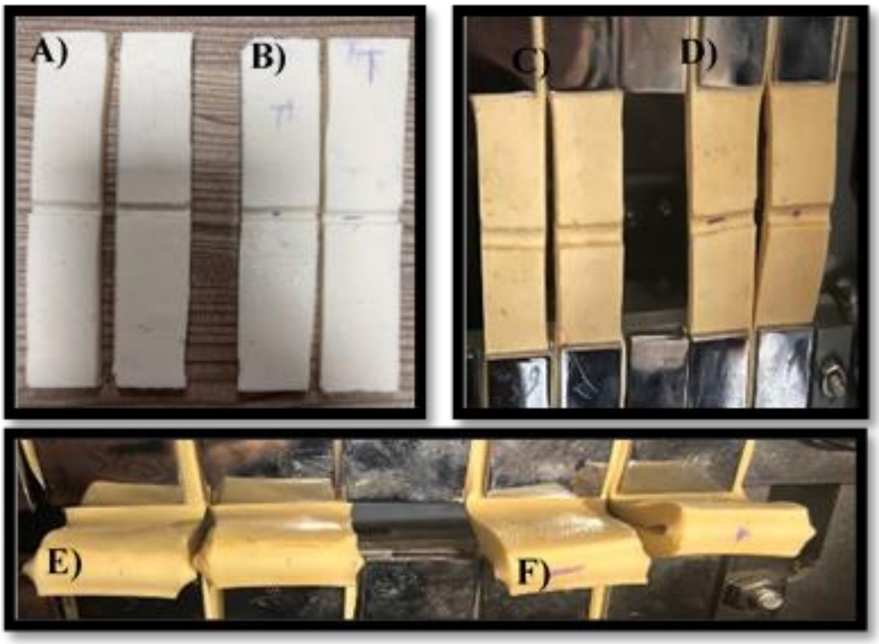

Fig.9: Test Samples of Fatigue (A, B before test C, D, E, F during the test)

Fig.10: Fatigue device

Resilience test

Samples were prepared according to International Standard No. ASTM D2632 for the preparation, examination, and comparison of results between two blends,[16]. as shown in Fig. 11.

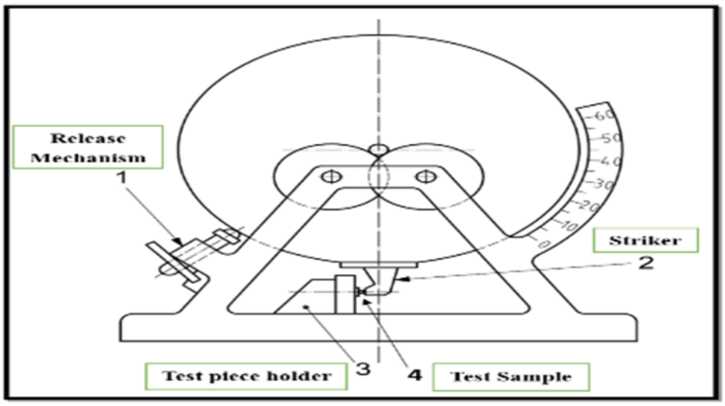

Fig.11: Illustration of resilience[16]

Swelling test

Two samples were taken from the above-mentioned mixtures with dimensions (1 thickness * 5 length) mm, then weighed and placed in liquid for a period of 20 hours at 25 °C, then extracted from water, dried, and weighed again to verify the swelling rate of the samples according to specifications ASTM D 3616 [17]. Related to specific gravity (S.G.), adopt the same hardness Mold while the specific gravity is checked on the density apparatus shown below as per ASTM D792 [18].

Fourier Transform Infrared Spectroscopy (FTIR)

The samples for FTIR analysis are prepared by mixing a small piece of each sample with KBr powder and then compressing it to form a tablet. Then Fourier transform infrared spectroscopic measurements were carried out using an 8400S FTIR-Perkin-Elmer spectrophotometer in the College of Materials at the University of Babylon. This test was carried out on (pure SR, pure PU, pure TEOS, {SR+PU}, and {SR +Pu + TEOS}) [19] .

It was conducted Scanning Electron Microscopy scans a surface to project an image by electron beam focusing. After the sample is plated with gold, it could be utilized to gain information about the composition and topography of the surface when the electrons in the beam interact with the sample .

Results and discussion

Results of several tests employed in the current study shown in Table 1 and Fig. 12. Based on these results, an increase in the physical properties such as (Tensile, Tear, Compression, hardness, Fatigue, Resilience and Specific gravity) was observed, and a decrease in swelling after adding the crosslinker material (TEOS).

Table 1: All results of tested under consideration

Test	Tensile Strength KPa	Tear KPa	Comp. %	Hardness	Fatigue Cycle	Resilience %	Swelling %	S.G g/cm^3
Grafted	0.5	12	3.2 %	32.7	3,100,023	22 %	3.4 %	1.109
ungrafted	0.27	11	5.5 %	25	1,587,120	19.5 %	10 %	1.001

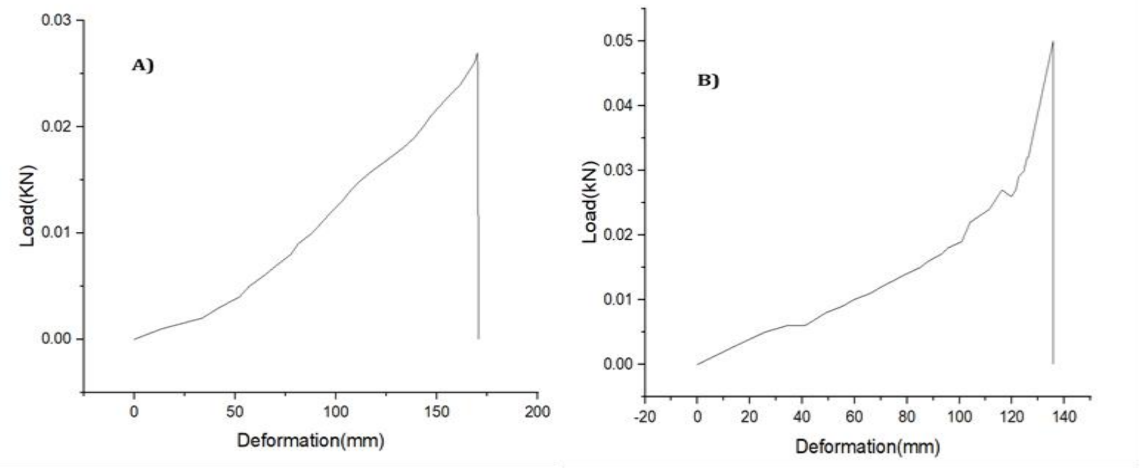

Fig. 12: The stress-strain diagram of sample (A) before adding (TEOS) and sample (B) after adding (TEOS).

Significant improvements can be noticed in several metrics such as tensile strength, tear resistance, compression strength, hardness, fatigue resistance, resilience, and specific gravity, highlighting the efficacy of TEOS in reinforcing the material. Of particular significance is the considerable decrease in swelling subsequent to the integration of tetraethyl orthosilicate (TEOS), which suggests an enhanced ability to resist the absorption of fluids and maintain dimensional stability. This attribute is crucial for specific applications[20]. The aforementioned enhancements arise as a result of the interplay of the Silane-Coupling Agent, polymer (SR), and filler material (Pu), which culminate in the formation of a resilient internal network. The chemical interactions are validated by the FTIR spectroscopy depicted in Figure 14. Moreover, this contact imposes limitations on the mobility of polymer chains and increases the density of the material, as illustrated in Figure 13. Consequently, this leads to a reduction in gaps and an improvement in the resistance to cracking. In summary, the incorporation of TEOS as a crosslinker material has been found to greatly improve the physical characteristics of the material[21].This improvement is attributed to the chemical interactions facilitated by the Silane-Coupling Agent, which have been confirmed by the use of FTIR analysis. Enhancing material performance and durability through increased density and decreased swelling is of utmost importance, as it has significant implications for the advancement of materials in various applications.

Moving to findings via scanning electron microscopy (SEM), it's noticed from Fig. (13b) the presence of pores and the lack of complete miscible of the phase, and this confirms the lack of interaction between the components of the mixture, which leads to a decrease in density and an increase in porosity, as well as areas of weakness in the sample and fewer mechanical properties. It's also noticed from Fig. (13b) that the material is miscible in one phase, and this confirms the presence of a chemical interaction between the materials of the blend and the decrease in porosity, which makes the sample have better mechanical properties.

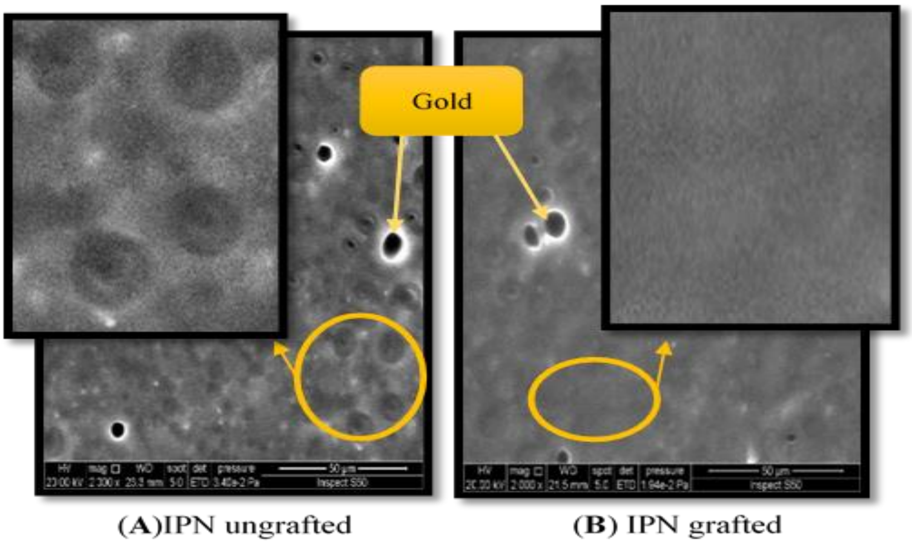

Fig. 13: SEM Test A (SR +PU), B (SR+ Pu +TEOS)

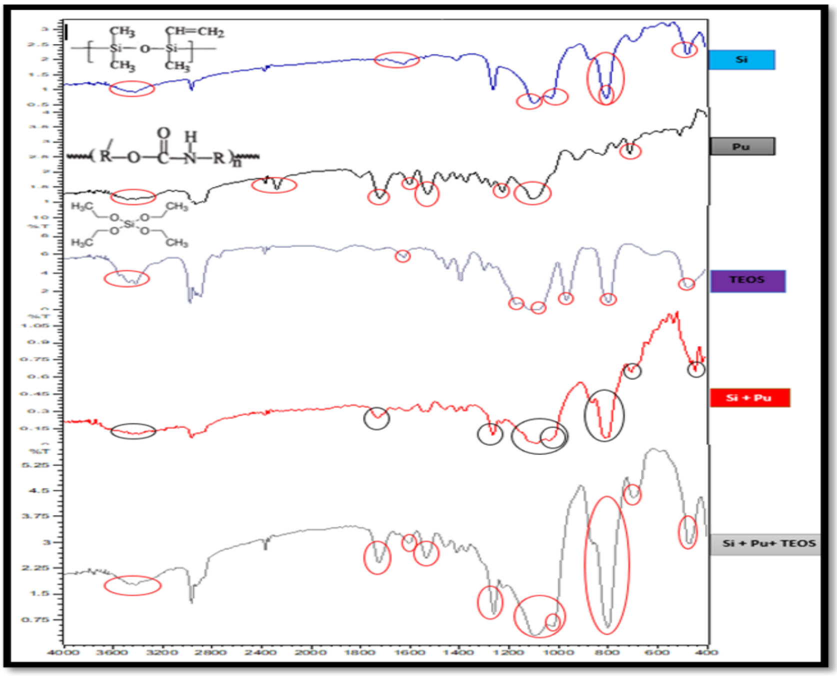

Fig. 14: FTIR for SR, PU, TEOS, (IPN Non-Grafted), and (IPN Grafted)

Table 2: Characteristic peaks and structures of (SR, Pu, and TEOS)

Peak Position (Cm-1)	Structure	Description
3310-3500	–N–H	stretching vibration
1613	C=C	stretching vibration
1080	Si–o–Si	Ring
1020	Si–o–Si	Linear
750-890	Si–O	stretching vibration wide peak
800	Si–C, Si–o–Si	Stretching vibration strong absorption
2270	N=C	Isocyanate Group

1720	C=O	stretching vibration
1630	C–O	Bending
1528	C–N, N–H, C–C	-------
1220	C–N	Bending
1127	C–O–C	stretching
1060	C–O–C	H-Bond
700	C=O, C–O	Stretching, Deformation
1630	C–O	Bending
1050	C–O, Si–O–Si	-------
950	Si-OH	-------
460	O–Si–O	-------

Moreover, Fourier Transform Infrared Spectroscopy (FTIR) is a very effective analytical method that facilitates the acquisition of significant knowledge regarding the chemical composition of various substances. Figure 14 illustrates a graphic showcasing SR (Silicon Rubber), Pu (Polyurethane), and TEOS (Tetraethyl orthosilicate), whereby discernible peaks may be observed in the FTIR spectra. The observed peaks in the data suggest that each material possesses distinct chemical compositions and elemental elements. The presence of these distinctive peaks offers valuable insights into the chemical composition and bonding arrangements present in these substances. Additionally, the FTIR spectra of SR, Pu, and TEOS exhibit distinct peaks, which are comprehensively summarized in Table 2, providing a full picture of their respective relevance. Through a comprehensive analysis of these prominent peaks, it is possible to get significant insights on the chemical characteristics and prospective utilizations of these substances. The comprehensive investigation of Fourier Transform Infrared (FTIR) data not only enriches our comprehension of the substances under investigation, but also underscores the multifaceted nature and exactitude of FTIR spectroscopy as an analytical instrument.

It's noticed from the blend diagram (SR+Pu) that characteristic peaks for polymers (SR) and (Pu) appear without fusion of peaks, and no new peak appears. This indicates the presence of physical mixing in the blend diagram for IPN Non-Grafted. Also, it’s noticed an overlap in the same peaks in Fig. 14. In the diagram (SR+Pu+TEOS), it's noticed that there is no new peak that differs from the basic polymers of the blend because of the basic polymer components of the blend, which (Si) have bonds and peaks similar to those in (TEOS). But this does not mean that there is no chemical reaction. It's been noticed that the peaks characteristic of the group (TEOS) and (SR) have increased in intensity. It's also noticed the width of peaks for the bond (Si-O) and (Si-O-Si) as well as (C-O) in (Pu) and also in (TEOS). This is clear evidence of a chemical reaction between the components of the blend. Where (TEOS) acts as a link between (SR) and (Pu).

Conclusion

The present study undertaking focuses on the complex procedure of combining polymeric components to create a cohesive mixture with the goal of improving mechanical qualities. The importance of compatibility and bonding between the elements of these blends on their ultimate qualities, particularly mechanical and general physical attributes, has been widely acknowledged. The primary objective of this work is to investigate the relationship between compatibility and the interaction of chemical and physical bonding dynamics in the formation of a thermosetting polymeric mix. The empirical evidence obtained from our thorough investigation sheds light on a compelling storyline: the combination, referred to as IPN (Interpenetrating Polymer Network), demonstrates a strong integration of both physical and chemical interactions. The aforementioned discovery yields concrete outcomes, resulting in a significant enhancement in many mechanical characteristics. The tensile strength exhibits a notable increase of 0.5 kilopascals, while the tear strength demonstrates a noteworthy rise of 12 kilopascals. Simultaneously, the fatigue endurance expands significantly to an incredible 3,100,023 cycles. In addition, a noticeable decrease in the swelling ratio is seen, reaching a minimal value of 3.4%. To substantiate these observations, Fourier Transform Infrared Spectroscopy (FTIR) provides supporting evidence for the presence of significant chemical linkages, particularly crosslinks, between the constituents of this composite, namely SR (Styrene Rubber) and PU (Polyurethane), facilitated by TEOS (Tetraethylorthosilicate). Simultaneously, the utilisation of Scanning Electron Microscopy (SEM) sheds insight on the gradual reduction of micro-gaps, which are widely acknowledged as a critical weakness in many mechanical properties. Hence, it is indisputable that the incorporation of grafted tetraethyl orthosilicate (TEOS) material elicits a profound impact on the physical characteristics of the interpenetrating polymer network (IPN) mix, namely in the case of styrene-butadiene rubber (SR) and polyurethane (PU), through the promotion of a complex network of intermolecular connections. This process results in an increased density, effectively closing the gaps between the different components of the blend. The aforementioned findings are supported by the insightful examinations conducted using Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM), which collectively present a compelling depiction of a synergistic effect that enhances the mechanical and physical properties of the polymeric mix.

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