

EFFECT OF QUAIL EGGSHELL FILLER ON THE ABRASION RESISTANCE AND THERMAL DEGRADATION OF ROOM TEMPERATURE VULCANIZING SILICONE RUBBER

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A B S T R A C T

Eggshells, which are mainly composed of calcium carbonate, have been considered as alternative filler in polymer composites to reduce cost and improve some of the material properties. One of the biofillers that has not yet widely studied for its potential use in rubber is quail eggshell. For this research study, the effects of quail eggshell filler on the abrasion resistance and thermal degradation of room temperature vulcanizing (RTV) silicone rubber were investigated. Samples with 0, 5, 10 and 15 wt. % quail eggshell filler were prepared through manual mixing and open molding process. Abrasion resistance test was performed based on ASTM D4060 while thermogravimetric-differential thermal analysis was utilized to examine thermal degradation. Results revealed that the abrasion resistance of silicone rubber increases as the eggshell filler content rises from 0 to 15 wt. %. The silicone rubber with 15 wt. % filler achieved the best abrasion resistance with a mass loss of about 44.7 % less than the sample without filler. For the thermal analysis, the sample with 15 wt. % eggshell filler achieved a higher peak degradation temperature of about 605.61 °C as compared to the plain sample (511.25 °C) but the latter appears to resist thermal degradation better at high temperatures.



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1. INTRODUCTION

The remarkable properties of silicone rubber such as superior resistance to heat, outstanding electrical insulation capability and excellent weatherability made this elastomer a popular choice in various applications.

In the automotive industry, silicone rubbers are used as sealants, gaskets, coverings and the like. Moreover, silicone rubbers can serve as mold materials for plastic resins, waxes and even alloys with low melting points (Shit & Shah, 2013). Another use is in the electrical parts, where the silicone rubber composites can be

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applied in high voltage insulations (Khan et al., 2018). Interestingly, due to the biocompatibility of silicone rubbers, they can also be used as implantable devices in the biomedical field (Cho et al., 2021). Likewise, silicone rubbers can be used as materials for food contact like baking mold and packaging (Liu et al., 2021). Fascinatingly, due to the characteristics and properties of these elastomers, it can also be applied as soft actuators or components in robots (Xavier et al., 2021). These wondrous applications of silicone rubbers make it a versatile and important material to study.

As one of the types of elastomers, silicone rubber is made of silicon and oxygen together with other elements namely carbon and hydrogen (Naguib et al., 2024). Typically, silicone rubbers are often in one part or two-component system where additives can be mixed to enhance some properties or features. In the two-component polymer, the base part should be mixed with the curing agent at their prescribed mixing ratio at the required condition in order to cure the material.

Silicone rubber can be classified into various kinds. In terms of the state of its vulcanization process, it can be categorized as room temperature or high temperature vulcanizing silicone rubber. As for the vulcanization mechanism, the types are addition and condensation. Also, it can be classified based on its suitable use in the environment which include categories such as temperature resistant, weather resistant, corrosion resistant, etc. (Han et al., 2022).

Although rubber materials like silicone offer great characteristics in different sectors, there are still some drawbacks. Some of the weaknesses of rubbers include poor tear strength (Yan et al., 2015) and low wear resistance (Liang et al., 2021). These disadvantages can somehow affect and limit the performance of rubber materials in certain applications like in covering components and molding materials where they can be subjected to excessive abrasion and can be exposed to tearing actions. However, there are methods that can be applied which might possibly enhance the properties of rubbers while reducing costs. One technique is by incorporating filler materials. Some of the fillers used in rubbers include silica, carbon black, talc and calcium carbonate (Phuhiangpa et al., 2020).

Usually, filler materials can be grouped into three categories which are the reinforcing type, the semi-reinforcing type and the non-reinforcing type. Factors that can affect the performance of using fillers in polymer include filler size, form and interaction between the filler and the matrix material. An example of common reinforcing filler which could enhance the mechanical strength of rubber is carbon black (Longkaew et al., 2023).

As for calcium carbonate, it is a commonly used filler in rubber since aside from lessening the costs, the mechanical properties are not compromised (Jong, 2020). Calcium carbonate is white in appearance that can naturally be seen as primary component in some rocks like limestone and chalk. Likewise, it can also be found in the composition of shells of marine organisms or shellfish (He et al., 2023). Calcium carbonate can be obtained through the crushing and grinding process of minerals that contain high amount of this compound like limestone where its ground form can be utilized in polymers such as plastics and rubbers as well as in other industrial applications (Cui et al., 2015).

Alternatively, waste materials that contain high content of calcium carbonate which can be used as biofillers in silicone rubbers include snail shells (Tepsila & Suksri, 2018) and eggshells (Khaigunha et al., 2020). As for natural rubber, some of the biofillers being considered include starch and cellulose (Thomas et al., 2021). Fascinatingly, using biofillers in composites do not only reduce costs but can also improve certain material properties while providing some environmental solution.

Apparently, there are already research works that utilized chicken eggshells as filler in composites (Kamath & Chandrappa, 2020). However, one of the farmed bird eggshells that is not yet further studied particularly in its potential use as biofiller in silicone rubber is quail eggshell. In the Philippines, the quail egg is one of the popular street foods. Though the quail eggshell is just being left as waste, exploring its use as an alternative filler in silicone rubber might yield favorable results. Moreover, quail eggshell has a high calcium carbonate content (Tan et al., 2015). This could make it a potential candidate as biofiller in composites. Thus, the researchers of this study would like to investigate the effects of quail eggshell filler on the properties of silicone rubber.

The considered properties in this study are abrasion resistance and peak temperature of thermal degradation since silicone rubbers are used in some applications that can be subjected to excessive abrasion and intense heat. The abrasion resistance can be used to know how resistant a material is against wear and tear due to the friction developed by rubbing it with an abrasive. On the other hand, the peak degradation temperature can help in identifying the limitation of the heat application of the material. This is to prevent it from being exposed at higher temperatures beyond its limitation to avoid thermal decomposition of its main components. Hence, the main aim of this paper is to determine the effects of quail eggshell filler on the abrasion resistance and the thermal degradation of RTV silicone rubber.

2. RELATED WORKS

This section discusses some of the related research works that used eggshell as filler in polymers such as epoxy, high-density polyethylene and rubber materials. Similarly, it also contains related studies that applied other biofillers like snail shells and mussel shells in polymer composites.

2.1 Eggshell filler in polymers

Using eggshell as biofiller in polymers can improve some of its properties. In a related work (Munusamy & Kchaou, 2023), powdered eggshells were used in acrylonitrile butadiene rubber to create latex films with varying filler content. The composite with eggshell filler achieved better tensile properties than the acrylonitrile butadiene rubber. Likewise, increase resistances to swelling and chemical degradation have been attained due to the addition of the filler in the rubber material. These could be linked to the good eggshell filler and rubber matrix interaction.

Similarly, in a related research work (Abdel-Rahim & Mohammed, 2019), eggshell fillers were incorporated in epoxy matrix with varying filler content. An increasing pattern in hardness has been observed as the filler loading rises. On the other hand, the water absorption lessens as the filler content increases. The epoxy with 16 wt. % filler obtained the best mechanical properties.

In a related paper (Farm et al., 2022), powdered eggshells were used as filler in high-density polyethylene. Carbonised and untreated eggshells with varying fiber loading were incorporated in the recycled polymer matrix. Improvement on the flexural strength and moduli were achieved with the addition of the eggshell fillers. The composite with 30 wt. % carbonised filler obtained the highest flexural properties.

In a related study (Skórczewska et al., 2022), eggshell powder served as the filler in poly(vinyl chloride) with varying filler amount. In terms of thermal stability, the filler helped in improving the decomposition temperature and increasing the stability time. As for the tensile properties, the modulus increases as the eggshell amount rises but the tensile strength reduces.

Likewise, in a related study (Khaigunha et al., 2020), silicone rubber has been incorporated with eggshells as filler which revealed enhancement on properties namely erosion and tracking resistance.

Apparently, with the related studies discussed above, eggshells can be used as an alternative filler in polymer composites which could potentially improve some mechanical properties, lessen water absorption, enhance thermal stability, increase resistances, etc.

2.2 Other shell filler in polymers

Aside from eggshells, sea shells also consist of calcium carbonate. Thus, these materials can be used as biofiller in polymers as well. In related research (Norazlina et al., 2015), seashells were used as filler in natural rubber. Varying particle size of 40 phr filler loading in natural fiber were tested. It was found out that as the particle size becomes larger, the hardness increases. Similar trend was observed in the tensile modulus. As for the tensile strength, the seashell filler size of 355 μm achieved the highest value.

In a research study (Tepsila & Suksri, 2018), snail shells were used as filler in silicone rubber. Varying filler percent and particle size were considered. Improvement on electrical properties as insulator have been attained due to the addition of the snail shell filler. Also, larger size of filler achieved better dielectric strength and constant. However, higher filler content led to a decrease in mechanical properties.

In a related work (Gigante et al., 2020), mussel shells were used as filler in a biocomposite of polylactic acid and poly(butylene adipate-co-terephthalate). The elastic modulus of the composite improves slightly as the amount of mussel shell powder increases while small decrease in strength was observed.

Like eggshells, using waste shell of marine organisms as biofiller in polymers can also enhance some of its mechanical properties. Although excess or higher amount of filler loading might lead to a decrease in value of some properties.

3. METHODS

This section of the paper discusses the materials used and the processes involved in the preparation of the specimen mold, the quail eggshell filler and the silicone rubber samples. It also includes the test methods used for the abrasion resistance and the thermogravimetric analysis.

3.1 Materials

The main materials used in this research are quail eggshell and RTV silicone rubber. Fresh quail eggs were bought from a local supermarket. The quail eggs were boiled in water and their eggshells were carefully removed. The waste eggshells were collected to be prepared as the filler material. On the other hand, a two-component liquid RTV silicone rubber was purchased in a local store. It is a platinum cure and food grade silicone rubber that has a mixing ratio of 1:1 by weight. The silicone rubber served as the matrix material.

3.2 Specimen Mold Fabrication

To create samples of RTV silicone rubber with quail eggshell filler, a mold material is needed. The 3-D design of the negative mold for the specimen testing was sketched using SolidWorks. It is rectangular in form that has a total length of 9.5 inches, a width of 5 inches and a height of 1/2 inch. The mold can accommodate 2 pieces of square shape specimens with dimensions of 4 in x 4 in x 1/8 in for the abrasion resistance test. Three pieces of mold were 3-D printed using PLA plastic as the material so about 6 specimens can be made per batch. Figure 1 shows the 3-D printed mold used for the specimen preparation.



Figure 1. 3-D Printed Mold for Specimen Preparation

3.3 Quail Eggshell Filler Preparation

The quail eggshells were washed with tap water to remove dirt and particles. The eggshells were then partially crushed and submerged in water so the remaining membrane in the eggshells can be manually removed in an easier manner. The eggshells were then sundried to get rid of excess moisture. After sun drying, the quail eggshells were pulverized using an electrical grinder and then stored in a clean container. The approximate size range of the crushed eggshells ranges from about 75 μm to 250 μm since the powdered sample passes through sieve no. 60 while most were retained in sieve no. 200. No chemical treatment was applied in the crushed eggshells. Figure 2 shows the partially crushed and the powdered quail eggshells.

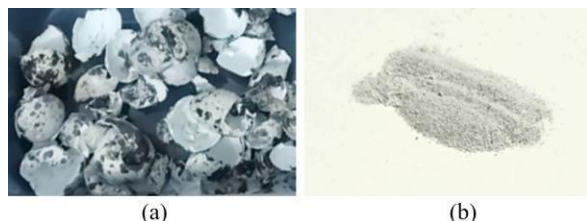


Figure 2. Quail eggshells: (a) partially crushed and (b) powdered

3.4 Specimen Preparation

The parts A and B of the liquid RTV silicone rubber was first manually mixed in a beaker with a weight ratio of 1:1 as recommended in the product description. The powdered quail eggshells were poured and then mixed in

this liquid mixture for about 5 minutes to disperse the eggshell filler in the matrix. The mixing of the components was done manually using a stirrer. Figure 3 shows the mixing process of the liquid silicone rubber and the quail eggshell filler. The mixture was then poured in the 3-D printed molds. The silicone rubber specimens were cured for about 24 hours at room temperature. After curing, they were carefully removed from the molds and then stored in plastic containers.

Silicone rubber specimens with varying weight percent composition of 0 %, 5 %, 10 % and 15 % quail eggshell filler were prepared which were labeled as S0, S5, S10 and S15, respectively. Only the filler loading mentioned was considered in the study so the effect of particle size was excluded in the scope due to time and cost limitations. Table 1 presents the composition of the sample types used in this research. For sample S0, only parts A and B of the silicone rubber were mixed. This specimen served as the baseline for evaluating if the quail eggshells in the other samples has helped improved its property. On the other hand, for preparing samples S5, S10 and S15, the appropriate weight of the quail eggshell filler and the liquid silicone rubber were measured using a gram scale before mixing these components to ensure that their corresponding weight % as shown in table 1 were followed.

Five pieces per sample type were prepared. Three pieces per specimen label were allotted for the abrasion resistance test while one specimen for the thermal analysis and the remaining as an extra piece. Figure 4 shows the samples of the cured silicone rubber with quail eggshell filler of 0 %, 5 %, 10 % and 15 % by weight.



Figure 3. Liquid silicone rubber with quail eggshell filler: (a) before mixing and (b) after mixing



Figure 4. Cured silicone rubber specimens S0, S5, S10 and S15 (from left to right)

Table 1. Composition of silicone rubber specimens with quail eggshell filler

Sample Label	Quail Eggshell (wt. %)	Silicone Rubber (wt. %)
S0	0	100
S5	5	95
S10	10	90
S15	15	85

3.5 Abrasion Resistance Test

The silicone rubber specimens were brought to a material testing laboratory where the test method used for the abrasion resistance test was ASTM D4060 adapted. Calibrade H-18 was used as the abrasive wheel. The number of cycles per load is 500 cycles at 500 grams. Teledyne Taber 5130 Abraser was used as the instrument for the abrasion resistance test. The conditioning atmosphere has a temperature of 23 ± 2 °C and a relative humidity of 50 ± 5 %. Three specimens were tested per sample type (i.e., S0, S5, S10 and S15). The mass of the specimens before and after the abrasion resistance test were measured using an analytical balance and the difference in mass was recorded. The abrasion resistance was expressed in terms of the weight loss of the samples in milligrams. The average value and the standard deviation of the three trials were computed. The sample that obtained the lowest mean value of the weight loss will serve as the specimen with the best abrasion resistance.

3.6 Thermogravimetric Differential Thermal Analysis (TG-DTA)

Specimen S0 and the specimen with the quail eggshell filler that obtained the best abrasion resistance were subjected to TG-DTA. Only these two specimens were tested due to budget constraint. The specimens were brought to a material testing laboratory for thermal analysis. The TG-DTA was done using Perkin Elmer STA 6000 as the instrument. The temperature range applied was from an initial temperature of 30 °C to a final temperature of 900 °C at a rate of 10 °C /min. Nitrogen was used as the gas atmosphere for the TG-DTA at a rate of 20 mL/min.

Three curves were plotted in excel spreadsheet based from the data recorded by the test instrument. The weight (%) of the sample versus the temperature (°C) represents the thermogravimetric analysis (TGA) curve. The first derivative of the weight with respect to time (%/min) versus the temperature represents the derivative thermogravimetric (DTG) analysis curve. On the other hand, the differential thermal analysis (DTA) curve relates the heat flow (mW) and the temperature. The peak

temperatures of the DTG and DTA curves were determined as well as the significant weight losses that occurred in the TGA curve. At the final temperature, the percent residue was also measured.

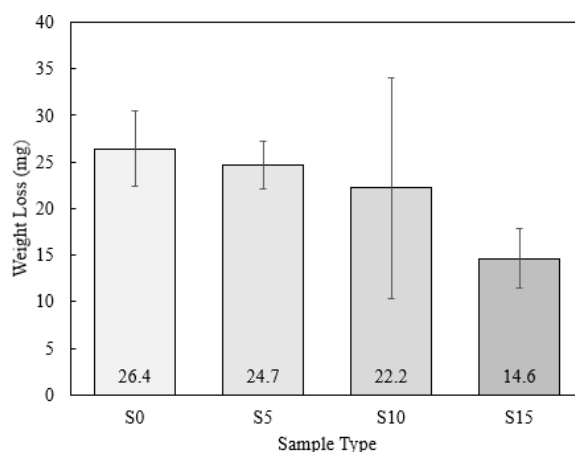
4. RESULTS AND DISCUSSIONS

This section presents the results obtained for the abrasion resistance test and the TG-DTA. It also includes discussions on the findings of the study.

4.1 Abrasion Resistance

Figure 5 shows the mean weight loss measured in the abrasion test of the specimens. The error bar represents the standard deviation. On the other hand, the measured weight loss per trial of the abrasion resistance test was shown in table 2. A lower weight loss indicates a higher resistance to abrasion.

Based from the results, S0 obtained the highest average weight loss with a value of 26.4 mg which means that it has the lowest abrasion resistance among the samples. While on the other hand, S15 has the lowest average weight loss having a value of 14.6 mg. As for specimen S5, it obtained a mean weight loss of 24.7 mg. As for S10, the average weight loss is 22.2 mg. Based from these results, the specimens with quail eggshell attained better abrasion resistances than the plain silicone rubber.

**Figure 5.** Abrasion resistance of silicone rubber specimens**Table 2.** Weight loss measured per trial in the abrasion resistance test of the specimens

Trial	S0 (mg)	S5 (mg)	S10 (mg)	S15 (mg)
1	24.9	26.4	35.8	12.4
2	31.0	21.8	15.7	18.3
3	23.3	25.9	15.1	13.2
Average	26.4	24.7	22.2	14.6
Standard Deviation	4.06	2.52	11.78	3.20

It can be noticed in figure 5 that the mean weight loss of the silicone rubber decreases as the quail eggshell filler loading increases from 0 to 15 wt.%. This means that the addition of quail eggshell filler in RTV silicone rubber increases the abrasion resistance. This enhancement could be due to the dispersion of the eggshell particles on the surface of the silicone rubber that lessens the contact area of the soft silicone matrix to abrasive actions. Similar observation was obtained in a related study where the wear resistance of natural rubber increases as the waste eggshell filler loading becomes higher (Ramesh et al., 2018).

Specimen S15, which has the highest filler loading, has the best abrasion resistance among the sample types. S15 only incurred a mass loss of about 44.7 % less than S0. This could also be due to the better dispersion of the eggshell at higher filler loading in the silicone rubber matrix. Likewise, the quail eggshells at higher filler loading might have filled the air spaces much better inside the silicone rubber which led to a higher abrasion resistance.

The standard deviation calculated for specimens S0, S5 and S15 are 4.06, 2.52 and 3.20, respectively. From trials 1 to 3, S0 obtained corresponding weight loss of 24.9 mg, 31.0 mg and 23.3 mg. For the case of S5, the weight loss for the trials are 26.4 mg, 21.8 mg and 25.9 mg. For S15, the respective values of the mass loss from trials 1 to 3 are 12.4 mg, 18.3 mg and 13.2 mg. The small values of the standard deviation obtained for these three sample types could indicate consistency in the trials.

As for S10, a value of 11.8 for standard deviation was computed where trials 1 to 3 obtained corresponding weight loss of 35.8 mg, 15.7 mg and 15.1 mg. This difference of the first trial from the other two trials of specimen S10 could be linked to the manual sample preparation where the filler material might not be properly dispersed in the silicone rubber during the mixing process as compared to the other specimens that have been made. Also, another factor that might contributed to the deviation is the particle size range of the powdered quail eggshells used in the sample preparation. Nevertheless, the decreasing pattern of the average weight loss as the quail eggshell filler content increases from 0 wt. % to 15 wt. % in silicone rubber is noticeable.

4.2 TG-DTA Results

Table 3 shows the peak degradation temperatures and the weight loss of samples S0 and S15 obtained in the thermal analysis. The peak degradation temperature indicates the temperature at which a high rate of weight loss has occurred in the sample. The graphical representations of the results namely TGA curve, DTG curve and DTA curve are presented in figure 6, figure 7 and figure 8, respectively.

As shown in table 3 and figure 7, S0 obtained DTG peak temperatures of 511.25 °C and 619.80 °C. These DTG peak temperatures correspond to the thermal degradation of RTV silicone rubber. As for S15, the first peak temperature recorded is 605.61 °C which is also due to the degradation of the silicone rubber matrix. The second peak degradation temperature for S15 is 763.98 °C, which is associated with the thermal degradation of the quail eggshells. In a related study, for the case of chicken eggshell, it starts to undergo thermal decomposition at around 700 °C (Awogbemi et al., 2020).

Comparing the results of the samples, the first peak temperature of S15 is higher than S0 (511.25 °C) which might be due to the influence of the quail eggshell. This could be due to the higher degradation temperature of the eggshell than silicone rubber. However, in terms of the weight loss as seen in figure 6, it appears that S0 has better resistance to thermal degradation than S15 at temperatures above 400 °C. Moreover, the total weight loss for S0 from 30 °C to 700 °C is only 50.34 % as compared to S15 having a value of 56.13 %. This could be linked to the poor adhesion of the quail eggshell filler to the RTV silicone rubber matrix since no chemical treatment has been used to aid their interaction. Nonetheless, the behavior of the TG curve for the two samples is almost the same at temperatures below 400 °C. As for the residue at the final temperature of 900 °C, S15 obtained a lower value of 37.46 % than S0 with a value of 46.95 % which is due to the decomposition of the quail eggshell that reduced the remaining content.

For the DTA results, a peak temperature of 496.86 °C is recorded for S0 while 596.87 °C and 758.50 °C for S15. These peak temperatures are associated with the endothermic events that occurred in the samples upon application of heat. These are mainly due to the thermal decomposition of the components. The DTA peak temperatures of 496.8 °C for S0 and 596.87 °C for S15 are due to the thermal degradation of the silicone rubber matrix. On the other hand, the DTA peak temperature of 758.50 °C is due to the thermal decomposition of the quail eggshell filler. Moreover, it can be noticed in table 3 that these DTA peak temperatures agree with the values obtained for the DTG peak temperatures.

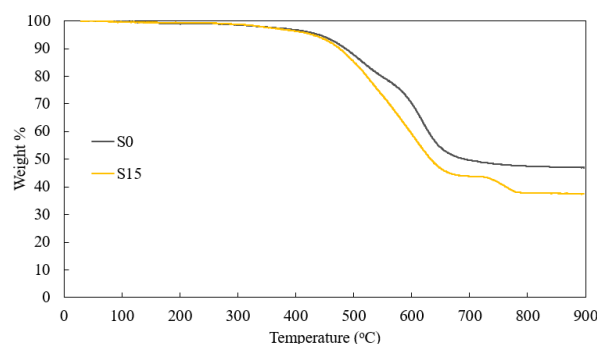
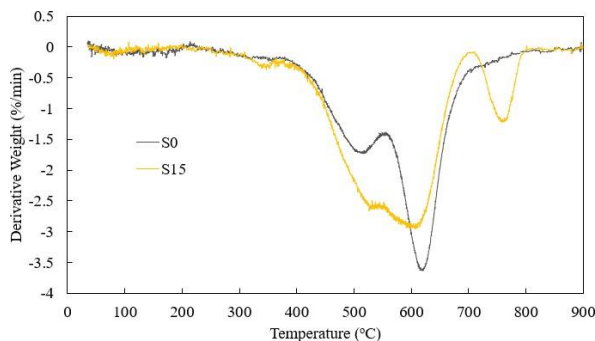
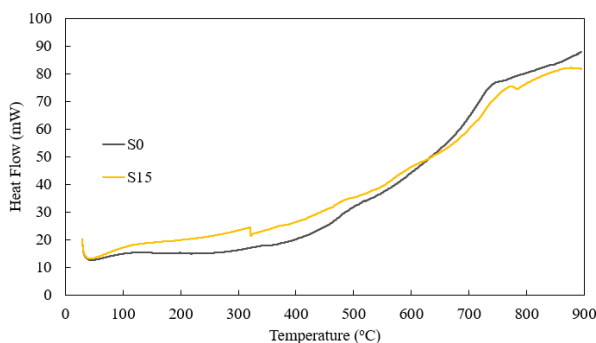


Figure 6. TGA curve of S0 and S15

Table 3. Peak degradation temperatures and weight loss of S0 and S15

Sample	Temperature Range (°C)	DTG Peak Temperature (°C)	DTA Peak Temperature (°C)	Weight Loss (%)	Residue (%)
S0	30 – 700	511.25	496.86	50.34	46.95
		619.80	–		
	700 – 900	–	–	2.71	
S15	30 – 700	605.61	596.87	56.13	37.46
	700 – 900	763.98	758.50	6.41	

**Figure 7.** DTG curve of S0 and S15**Figure 8.** DTA curve of S0 and S15

5. CONCLUSION

The abrasion resistance and thermal degradation of RTV silicone rubber with quail eggshell were investigated in this paper. Based from the results, the abrasion resistance of silicone rubber enhances as the eggshell content increases from 0 to 15 wt. % which could be linked to the filler dispersion that reduces the contact area of silicone to abrasion. Among the samples, S15 obtained the best abrasion resistance with a mass loss of about 44.7 % less than S0. As for the thermal analysis, S15 achieved a higher peak degradation temperature of 605.61 °C as compared to S0 (511.25 °C) which might be due to the higher decomposition temperature of the eggshell. However, S0 appears to resist thermal degradation better than S15 which could be associated to the poor filler-matrix interaction due to lack of chemical treatment. Nevertheless, with the improvements attained, quail eggshells can be considered as an alternative filler in silicone rubber where its composite can potentially be applied as mold material for plastics. For future works, it can be recommended that more tests on material properties of the RTV silicone rubber with quail eggshell filler could be conducted. Particle size range of the quail eggshell filler could also be considered as another factor to evaluate aside from the filler loading.

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