



## Utilization of Silica Fume and Sodium Hydroxide in Treated Crumb Rubber for Cement Mortar

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### ABSTRACT

Cement mortar offers an excellent replacement for materials such as fine aggregate with tire rubber waste in the form of crumbs. It provides excellent environmental and technical benefits to concrete production using recycled materials. As such, it contributes to the sustainable development of the construction industry. This paper mainly emphasizes the strength of untreated and treated crumb rubber from waste tires in cement mortar. Crumb rubber that has been pre-treated with Silica fume (SF) and sodium hydroxide (NaOH) to improve the strength of the mortar mix. This research used a cement-aggregate mix ratio of 1:4 and a water-cement ratio of 1:2. Five different percentages of fine aggregate replacement (0, 3, 6, 9, and 12%) was selected. The compressive strength, density, and water absorption of the mortar were measured at 28 days to find optimum strength. The compressive strength of the cement mortar mixed with treated crumb rubber showed significantly higher values, with an increase of 92% for 12% treated crumb rubber by sodium hydroxide (TN12) compared to untreated crumb rubber. The density value of the mortar cube mixed with treated crumb rubber decreased when the percentage of replacement for treated crumb rubber increased. In the application of roof tiles, lower density values provide an advantage for workability during installation. For water absorption, the treated crumb rubber contributes to a lower percentage of water absorption (acceptable until 6% for SF and 9% for NaOH) compared to the control sample as untreated crumb rubber. Therefore, a mixture of mortar with treated crumb rubber, especially NaOH solution, is better than the untreated crumb rubber specimen.

## 1. Introduction

Now, the increasing population causes many new building structures to be needed in rising countries. However, it is hard to keep up with the rising costs of building supplies. Traditional

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construction materials such as cement, sand, and stones are crucial in producing concrete and mortar. Due to the rising cost also, researchers are exploring the possibility of utilising industrial, construction, and agricultural waste as building materials. The main components in mortar are fine aggregates, Portland cement (limestone), and water. The quality and strength of mortar might vary significantly depending on the mixing proportion. Mortar has been used in various applications in the construction industry and is considered very affordable. Studies on the utilisation of rubber content in a replacement for aggregates have been ongoing for the past decade as part of the sustainable solution to dispose of waste tyres that are accumulating globally [1]. Waste tyres contribute to a significant portion of solid waste, a global environmental challenge. It is estimated that around 8 billion tyres are thrown out around the world. According to the Malaysian Ministry of Housing and Local Government's National Solid Waste Management Department's yearly report, around 0.3 million tonnes of scrap tyres are created [2]. It was predicted that this number would rise at a rate of 5% per year, in line with the rise in automobile usage. The amount of scrap tyres generated by 2025 is projected to be around 0.4 million tonnes [3]. Tyres is commonly burned to extract materials such as steel or fuel, which could harm humans and the environment [4].

In a good way, rubber can act as a binder or be used to replace fine and coarse aggregates in concrete and mortar. Crumb rubber particles have been extensively studied as filler ingredients in conventional concrete and mortar [5]. Recycling used tyres in construction is also an environmentally friendly way to reduce waste and save money [6]. Using rubber reduced carbon dioxide emissions and improved the environmental sustainability of any engineering cementitious composite [7, 8]. The large size of the construction industry provides a large avenue for recycling waste tyre materials, and hence, should be given utmost attention. Laboratory investigations have retrieved that the substitution of waste tyre particles in concrete and mortars substantially increases their toughness, impact resistance, energy absorption, plastic deformation, and fracture criteria of concrete [9]. However, previous investigations revealed several weaknesses incorporated with strength properties when using waste tyre particles [10,11].

In a mortar, weak adhesion between rubber and cement was found to lead to a considerable decrease in the strength of mortar that incorporates recycled tyre crumbs. The lower specific gravity of tyre crumbs also resulted in the reduction of the unit weight of mortar. However, the reduced compressive strength has hindered the use of recycled tyre crumbs in concrete or mortar. The loss of strength is mainly due to poor adhesion between recycled tyre crumb and cement mortar because recycled tire crumb is hydrophobic (water-repelling) in nature. Some efforts have been made to improve the strength properties by proper surface treatment of the waste tyre particles to develop the connection strength between the surface of the waste tyre and the cement [12]. The surface pre-treatment should roughen the tyre particles, which provide a better bond, thus, yield higher strength properties [13]. Successful surface treatment is achieved using waste organic sulphur compounds [11], partial oxidation of the crumb tyre [14] and oil palm fruit fibre (OPFF) [9,15]. The treatment of tyre crumbs using sodium hydroxide (NaOH) solution has also been successful in increasing the adhesion between cement and crumbs by engraving the surface of the crumbs to give increased strength [16-18].

Most of the previous research studies focused on concrete mix compared to the mortar needed in roof tile applications. Ameri *et al.*, [19] reported the partial replacement of copper slag mixed with crumb rubber aggregates treated in alkali-activated slag mortar. They used 5, 10, and 15% (by volume) treated crumb rubber in replacing alkali-activated slag mortar and showed a decrement in compressive strength as the percentage of crumb rubber replacement increased. Onuaguluchi [20] highlighted on pre-plating of crumb rubber in limestone powder and mixing with silica fume to perform rubberized cement mortar. They also used 5, 10, and 15% by volume of pre-plating crumb

rubber as a replacement for fine aggregate and demonstrated that the strength of the mortar reduced when the percentage of rubber replacement reached 15%. However, However, the use of a smaller percentage difference is very helpful to know the sensitivity of the data obtained.

This paper focuses on using waste tires in mortar by pre-treatment of rubber crumbs for use in roof tiles. Pre-treatment was used on crumb rubber in concrete because the strength of the mortar is reduced compared to mortar without a crumb rubber mixture. Therefore, surface pre-treatment was used to improve the mechanical performance of the specimen. However, fewer studies were found in the literature on the effects of treated crumb rubber with silica fume (SF) and NaOH solutions on mortar specimens. Therefore, this study was conducted to compare the performance of untreated and treated powdered rubber (various treatment solutions) as a partial replacement of aggregates in mortar specimens on physical strength including compression, density, and total water absorption.

## 2. Methodology

### 2.1 Material Preparations

The materials included in the mortar production are water, cement, fine aggregate (sand), and crumb rubber (untreated and treated) as partial aggregate replacement. The cement Type I Ordinary Portland cement (OPC) from YTL Cement, Johor, Malaysia, was used in this study as shown in Figure 1. The OPC was allowed to pass through a 300 mm opening sieve based on ASTM C150 [21] to separate the cement clinker before being kept in an airtight container to avoid contact with moisture in the air. This is because cement particles exposed to moisture can be hydrated and disrupt their pure condition. A mixture of mining and sand was used as fine aggregate helping to increase the tensile strength. A sieve analysis test was conducted to distribute the particle size of fine aggregate. The sieves used have size graduations of 0.075 to 4.75 mm, respectively. The coarsest sieve was placed at the top, while the finest sieve was placed at the bottom of the sieve analysis.



Fig. 1. Portland cement

Water is one of the primary raw materials needed to produce mortar. Because it will affect the hydration process of the specimens, the water must be clean and free of impurities. The water used to mix the mortar was compliant with ASTM C1602 [22]. Raw water can be derived from various sources, including mixed water, non-potable water, mixing water, and potable water, if the water does not include any harmful contaminants that could short- or long-term influence the cement's hydration process. Clean water was used in this experiment to mix with the mortar casting and a 1:2 water-cement ratio was applied. The crumb rubber was taken from a rubber factory at Parit Sulong,

Batu Pahat, Johor. The maximum size of the crumb rubber was 3mm. Figure 2 shows the particle of crumb rubber used in this research. Compared to sand, crumb rubber contains irregular circular forms while sand has irregular shapes. The untreated and treated crumb rubber was used as a partial replacement for fine aggregate. The crumb rubber particles were sieved between 0.075 and 4.74mm. The percentage of replacement used was 0, 3, 6, 9, and 12%.



Fig. 2. Crumb rubber

Figure 3 shows a NaOH was used as a surface treatment for the treated crumb rubber before being mixed with other raw mortar materials. The crumb rubber was soaked with 20% NaOH solution for 24 hours in a container and then rinsed with tap water to prevent any side effects to mortar. Safan *et al.*, [23] reported improved compressive, flexural, and tensile strength of concrete mixed with crumb rubber treated using NaOH. They concluded that 20% NaOH of pre-treatment gave a better performance. The pH of the water was checked until the pH became neutral (7.0). The total time for the removal of the NaOH solution from the rubber surface was around 30-45 min. After that, the crumb rubber was allowed to dry before it was used to mix. Cube mould or mortar with dimensions of 50 × 50 × 50 mm was used to cast the mortar specimens. The mould must be cleaned of dirt residues before using it. After that, the inner surface of the moulds was coated with grease for ease of demolding after the mortar had hardened.



Fig. 3. Sodium hydroxide

A package of silica fume (SF) used in this study is shown in Figure 4. Silica fume has been used as pre-treatment for crumb rubber particles to improve the strength of mortar specimens. Silica fume has been routinely used as a replacement for cement in concrete mixes to provide increased strength for concrete specimens. However, no results have been found against the use of silica fume as a pre-treatment for crumb rubber in mortar specimens. Silica fume particles with a median particle diameter of 0.1–0.3 microns. The crumb rubber has been soaked in a mixture of silica fume and water as a pre-treatment process. The pre-treatment process of crumb rubber was soaked for 24 hours in the treatment solution. Silica fume contains 85-95% of silicon dioxide ( $\text{SiO}_2$ ). Silica fume collected

from the manufacture of silicon and silicon carbide metal. The silicon dioxide ( $\text{SiO}_2$ ) released in the production process of these alloys undergoes oxidation and condenses in the form of very fine spherical particles of amorphous silica. Silica fume significantly improves the solids concentration, mechanical properties, and durability of cementitious materials due to its physical and chemical characteristics, which provide high reactivity with Portland cement hydration products [24,25].



Fig. 4. Silica fume

## 2.2 Treatment Process

Figure 5 shows the crumb rubber treatment process using two additives which are silica fume and sodium hydroxide solutions. When treated rubber is mixed into cement mortar, the cement hydration surrounding the rubber particles can be improved due to the weak alkali state created by the NaOH and silica fume solutions. Furthermore, the sodium hydroxide solution treatment increases the hydrophilicity of crumb rubber, lowering the porosity between crumb rubber and cement paste. As a result, rubber and cement paste adhesion could be improved. A 20% silica fume and 20% NaOH by water weight were used separately to make the pre-treatment solution on crumb rubber particles before the mixing process with cement and fine aggregate. Rubber tyre aggregates were soaked in the solution for 24 hours (at least 24 hours) according to ASTM C114-18 before being mixed into the mortar [26]. The crumb rubber was cleaned with clean water until the condition of the water approaches pH 7.0 after the treatment process with SF and NaOH solutions. Finally, the washing water was discarded, and the treated crumb rubber was allowed to dry at room temperature.



Fig. 5. Treatment of crumb rubber additive solution

## 2.3 Fabrication Process

The mortar mixing and curing procedures followed the standard laboratory practice. All processes of treatment, mixing, and curing were placed at Concrete Technology Workshop, Universiti Tun Hussein Onn Malaysia, Pagoh Campus. The raw materials include cement, sand (fine aggregate), and untreated/treated crumb rubber dry mixed. Manual mixing was used to mix all the materials



according to the mix design as shown in Figure 6(a). The mixture proportions were stirred together until the mortar mixture blended uniformly. Before that, for the treated crumb rubber, the particles were treated using two different solutions which are silica fume and sodium hydroxide for 24 hours and were dried before being used. Then, water was added to the mixture until the concrete appeared homogeneous and of the desired consistency, as shown in Figure 6(b). The mortar mixture was visually assessed to ensure the proportions were uniformly mixed. After completing the mixture, the inner mould surface was lubricated with a layer of oil before the cement mortar mixture was poured into the mould in three layers. Figure 6(c) shows rod tamping was used to compact the mortar cube specimens. To give a smooth surface flush, the outermost layer of the mortar was levelled off with a trowel to balance the slightly overfilled concrete on top of the mould. After the casting process was completed, all specimens of mortar with a size of 50 × 50 × 50mm (according to ASTM C109) were left for 24 hours after casting before being demoulded. Figure 6(d) shows that the cube sample was weighed after being demoulded [27]. Finally, Figure 6(e) shows all specimens of mortar ready to place in a curing tank (at Concrete Technology Workshop) for 28 days. The water tank used for the curing process was made of non-corroding material, and the water temperature inside the tank was kept between 25 and 28 °C.



**Fig. 6.** Process of (a)mixing raw materials, (b)mixing with water ratio, (c)compacting each layer using a tamping rod, (d)weighing cube sample, and (e)ready place in the curing tank

**Table 1**

Mix design for all specimens

Percentage of crumb rubber (%)	Untreated	Treated by SF	Treated by NaOH
0	Control sample (CS)	-	-
3	U3	TSF3	TN3
6	U6	TSF6	TN6
9	U9	TSF9	TN9
12	U12	TSF12	TN12

Five sets of mixed proportions for untreated and treated crumb rubber mortar with different solutions, including one controlled mix, were produced. For treated crumb rubber solution was divided into two (2) solutions with silica fume (SF) and sodium hydroxide (NaOH). The mixture without the crumb rubber particles (0% replacement) was set as the control sample (CS). The percentage of crumb rubber particle proportions were 3, 6, 9, and 12%, as shown in Table 1. The cement mortar mixture used a weight-to-weight ratio for water, cement, and sand (0.5:1:4). Every mixed proportion was conducted in three specimens due to finding the average compressive strength for each combination.

## 2.4 Mechanical Testing

Figure 7(a) shows the compression testing of the cement mortar specimens conducted at the Concrete Technology Workshop, Universiti Tun Hussein Onn Malaysia, Pagoh Campus based on the ASTM C109 standard [27]. The compression testing of all specimens was conducted using the AUTOMAX Pro compressive testing machine with a rate of load compression of 0.7 MPa/s as shown in Figure 47(b).



**Fig. 7.** (a) Mortar cube specimen, (b) Compressive testing machine

Three specimens in each mixed proportions were prepared for water absorption testing as complying with ASTM C1403-15 standard test method was used to determine density, absorption, and voids in hardened mortar. As a first step, the cube specimens were required to be submerged in water at a temperature between 15.6 °C and 28 °C for 24 hours. The specimen was taken out of the water and left to air dry after they had been immersed. A moist towel can be used to remove surface water from the specimens, and the weight of the specimens was recorded as saturated weight ( $W_{sat}$ ) using a weighing machine. The specimen was then dry in an oven for 24 hours as shown in Figure 8 and the oven-dry weight of the specimens was recorded as dry weight ( $W_{dry}$ ) after the saturated weight of each specimen had been recorded. It was then taken out of the oven the next day and allowed to cool to room temperature before being physically measured for mass.



**Fig. 8.** Water absorption testing

### 3. Results

#### 3.1 Compressive Strength

Figure 9 shows the physical strength of hardened crumb rubber mortar specimens with 0, 3, 6, 9, and 12% untreated and treated crumb rubber for a replacement of fine aggregate (sand) at 14 and 28 days. The compressive strength of the specimens increases with the increasing age of curing. The compressive strength for treated crumb rubber increases compared to untreated crumb rubber from 2.83 to 23.10 MPa at 14 days and 4.92 to 25.43 MPa at 28 days of curing, respectively. However, a downward trend was recognized with the increment of crumb rubber replacement. This behavior could be associated with inadequate binding between cement and crumb rubber instead of fine aggregate and cement paste. The significant drop in compressive strength also was governed by cement containing rubber is much softer than cement that hardens without rubber, and the crack quickly develops within the rubber under loading [28]. As mentioned by Senin *et al.*, [29], a significant rise in compressive strength from 3 to 5% replacement was contributed by the small size of rubber ash used as a gap filler. However, using more than 5% of rubber reduces the compressive strength due to the high rubber ash concentration in cement mortar specimens making it bouncier and elastomer. The combination of sawdust in concrete mortar as a partial replacement also decreases compressive strength with an increasing amount of sawdust concrete mortar [30]. Rubber has natural properties as an elastomer and provides a reduction in the compressive strength of the mortar when large amounts of crumb rubber are placed. The use of a small amount of crumb rubber content should be managed to maintain adequate compressive strength [31].

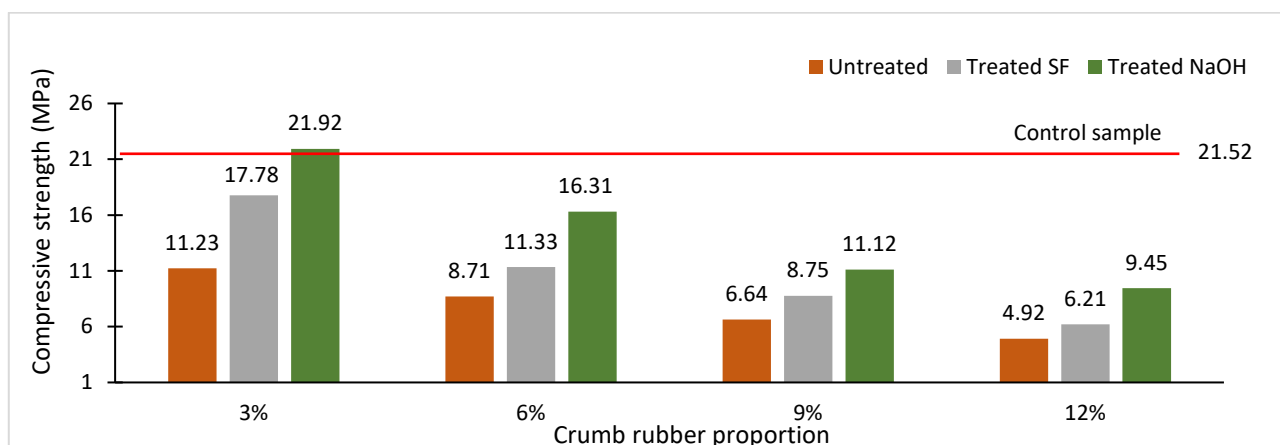


Fig. 9. Compressive strength of crumb rubber mortar at 28 days

The comparison between untreated and treated crumb rubber with similar crumb rubber proportions revealed a significant increase in compressive strength for treated crumb rubber. At 14 days of curing age, the highest increment recorded was at 3 and 6% of crumb rubber replacement. The compressive strength shows only a slight increase of 9 and 12 % of crumb rubber compared to the untreated crumb rubber specimens. Meanwhile, at 28 days age of curing, the compressive strength increases notably for 0 to 12% crumb rubber replacement. This finding was aligned with the findings from You's work, where increasing the curing age succeeded in increasing the compressive strength [32]. According to Safan [23], the increase in rubber and cement bonds allows the rise of compressive strength by increasing the surface roughness of the rubber particles on a microscopic scale. The lower porosity between crumb rubber and cement also contributes to the higher compressive strength. Apart from the amount of rubber and additive, the other factor that can affect



strength is the applied temperature on the specimen where the high temperature gives the contribution to the decrement of strength values [33].

Despite the increment of compressive strength contributed using sodium hydroxide in the treatment process of crumb rubber, an undeniable decrement trend was still observed with the increment of crumb rubber replacement. A similar finding was obtained by Girskas and Nagrockienė [34], who mentioned that the rise of rubber amount in the mortar would decrease compressive strength. However, optimum strength can still be achieved with an adequate amount of rubber. Overall, the compressive strength obtained ranged between 4.92 to 21.52 MPa and 9.45 to 25.43 MPa for untreated and treated crumb rubber, respectively. The specimens of crumb rubber treated with sodium hydroxide improved the compressive strength by up to 92% (12% of replacement) compared to the untreated crumb rubber.

### 3.2 The Density of Mortar

The density of the mortar specimens obtained at 14 and 28 days for 0, 3, 6, 9, and 12% crumb rubber is shown in Figure 10. In general, the addition of treated crumb rubber shows appreciable improvement in the density value of the mortar samples compared to the untreated crumb rubber. The 3, 6, 9, and 12% treated crumb rubber replacement shows a significant increase in mortar density compared to untreated crumb rubber. Aside from 9 and 12% crumb rubber replacement at 14 days of curing age, the increment was slightly lower compared to 28 days age of curing. The control specimen of 0% replacement of crumb rubber shows a slight decrease in the density of the specimen for both 14 days and 28 days of curing. Overall, the density value of the mortar samples decreases relative to the percentage of increase in crumb rubber replacement. The density of mortar cement also decreased with increased curing age.

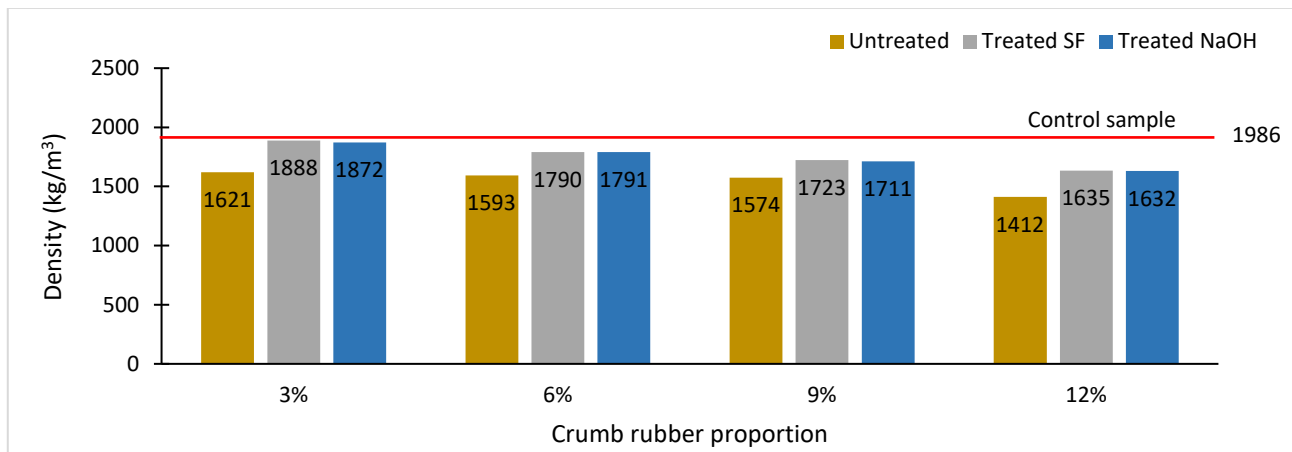


Fig. 10. Density of crumb rubber mortar at 28 days

### 3.3 Water Absorption

Figure 11 shows the average percentage (%) value of water absorption for mortar specimens with untreated and treated crumb rubber mortar at 28 days of age curing. The result showed that a lower water absorption rate value was recorded on specimens with treated crumb rubber compared to untreated crumb rubber. The crumb rubber treatment using sodium hydroxide solution allows for minimizing the value of water absorption for the mortar specimen up to 32% lower compared to untreated specimens. The water absorption value increases slightly as the percentage of crumb rubber replacement increases from 0 to 12% for all specimens tested. Mortar specimen with a high

percentage of crumb rubber has higher porosity, thus, a higher percentage of water absorption. Good quality mortar mixed required a percentage of water absorption well below 11% by mass, followed by ASTM C55-17. Other than porosity, the amount of water absorbed could also be affected by the kind of material, additives, temperature, and contact period. The water absorption was important as it can impact the mortar strength [35]. The percentage of water absorption for untreated and treated crumb rubber ranges from 9.12 to 17.52% and 10.46 to 11.81%, respectively.

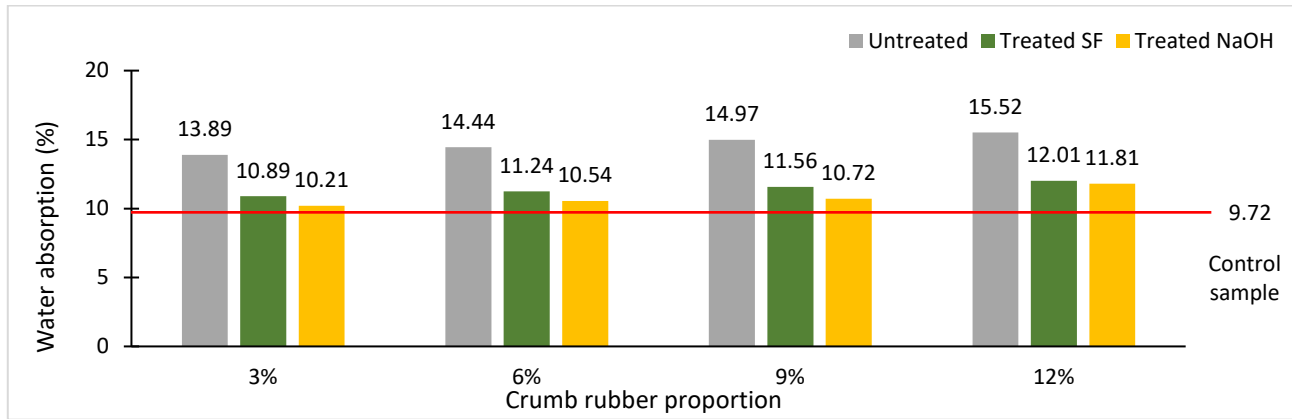


Fig. 11. Water absorption of crumb rubber replacement

### 3.4 Minimum Properties for Mortar Standard

Table 2 shows the overall experimental result in the current study for compressive strength, and water absorption test. ASTM C270 [36] is an available standard of mortar specimens to provide different mortar types for unit masonry. These standard addresses four types of mortar such as M, S, N, and O including the interior and exterior location of the building. These types are classified to the property specification requirements of mortar. Type M mortar is best suited for high-weight and below-grade applications such as foundations, retaining walls, and roadways. Meanwhile, type S mortar is often used for masonry foundations, manholes, retaining walls, sewers, and at-grade buildings such as brick patios and walkways. Type N mortar is the best choice for general use. Lastly, type O mortar is ideal for repointing and other related repair work on existing structures due to its uniformity and simplicity of application. From ASTM C270, the minimum compressive strength of mortar has been divided according to the type of location used in the building. The compressive strength of each type is 17.2 MPa (type M), 12.4 MPa (type S), 5.2 MPa (type N), and 2.4 MPa (type O). Based on ASTM C270 [36], the type of mortar for roof tiles application is classified as type S.

Table 2

The experimental result at 28 days age of curing

Test	Experiment result			Descriptions
	Untreated	Treated SF	Treated NaOH	
Compressive strength (MPa)	CS = 21.52			The minimum compressive strength for roof tiles application is type N mortar = 5.2 MPa
	U3 = 11.23	TSF3 = 17.78	TN3 = 21.92	
	U6 = 8.71	TSF6 = 11.33	TN6 = 16.31	
	U9 = 6.64	TSF9 = 8.75	TN9 = 11.12	
	U12 = 4.92	TSF12 = 6.21	TN12 = 9.45	
Water absorption (%)	CS = 9.12			ASTM C55-17 [37] specifies the maximum water absorption of regular (8%) and medium-weight masonry units (11.3%).
	U3 = 14.89	TSF3 = 10.89	TN3 = 10.21	
	U6 = 15.44	TSF6 = 11.24	TN6 = 10.54	
	U9 = 15.97	TSF9 = 11.56	TN9 = 10.72	
	U12 = 17.52	TSF12 = 12.01	TN12 = 11.81	

Based on Table 2, untreated crumb rubber proportions of CS, 3, 6, and 9% achieved a minimum compressive strength of roof tile mortar (types N). Meanwhile, 12% of untreated crumb rubber proportions were only applicable for mortar type O. For the treated crumb rubber of both solutions, all specimens were passed to achieve the minimum compressive strength for roof tiles application. This result shows an increase in the strength of the pre-treated specimen compared to the untreated specimen. In addition, the pre-treatment of NaOH is a better solution than the SF solution in increasing the compressive strength. Trung [37] has mentioned that with the addition of an additive coating to the composite, the flexural strength of the composite has improved. According to ASTM C55-17 [38], the maximum water absorption for the normal weight of the masonry unit is 8%, while for the medium weight of the masonry unit is 11.3%. The water absorption value obtained by mortar with treated crumb rubber was within the specified range. Meanwhile, for the mortar specimen with untreated crumb rubber (except the control specimen), the value of water absorption exceeded the required amount (based on ASTM C55-17). Overall, the treated specimen gave a better percentage for the water absorption value. The decreasing value of water absorption is very important for the application of the roof tile because it reduces the probability of leakage. Lastly, there is no specific standard exists for the density of mortar. However, in construction works such as roofing, the weight of a material affects workability and installation.

#### 4. Conclusions

The utilization of rubber particles as a partial replacement for fine aggregate in mortar is necessitated by the need to recycle abundant waste tires, which are non-biodegradable. However, the use of these materials often results from losses in strength properties. The losses were reduced by surface treatment using SF and NaOH solutions. A comparative study between the untreated and treated crumb rubber revealed a significant increase in compressive strength especially in NaOH solution with an increase of 92% (TN12). Referring to ASTM C270, all aggregate replacements using treated crumb rubber in both solutions achieved the minimum compressive strength specified for mortar N in roof tile application. Meanwhile, for untreated crumb rubber, only 12% partial replacement (U12) not achieved the minimum compressive strength of a similar mortar type. The adaptation of surface treatment using SF and NaOH solution also improved the average density and minimized the percentage of water absorption. The density value was also recorded for mortar cubes with untreated crumb rubber with a slight decrement as the percentage of treated crumb rubber increased. In the application of mortar products in construction, such as roof tile and brick, the lower density value was given advantages (workability). The lower percentage of water absorption corroborates an increase in overall compressive strength. Therefore, the outcomes showed that using treated rubber crumb to replace fine aggregate in the production of rubber mortar has been successful without affecting the compressive strength.

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