WARSAW UNIVERSITY OF TECHNOLOGY Index 351733
FACULTY OF CIVIL ENGINEERING
COMMITTEE FOR CIVIL AND WATER ENGINEERING
POLISH ACADEMY OF SCIENCES ISSN 1230-2945

DOI: 10.24425/ace.2022.141875
ARCHIVES OF CIVIL ENGINEERING
Vol. LXVIII ISSUE 3 2022

pp. 87 –105

Research paper

Crumb rubber geopolymer mortar at elevated temperature exposure

Ahmad Azrem Azmi[1], Mohd Mustafa Al Bakri Abdullah[2], Che Mohd Ruzaidi Ghazali[3], Romisuhani Ahmad[4], Ramadhansyah Putra Jaya[5], Shayfull Zamree Abd Rahim[6], Mohammad A. Almadani[7], Jerzy J. Wysłocki[8], Agata Śliwa[9], Andre Victor Sandu[10]

Abstract: Low calcium fly ash is used as the main material in the mixture and the crumb rubber was used in replacing fine aggregates in geopolymer mortar. Sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) which were high alkaline solution were incorporated as the alkaline solution. The fly ash reacted with the alkaline solution forming alumino-silicate gel that binds the aggregate to produce a geopolymer mortar. The loading of crumb rubber in the fly ash based geopolymer mortar was set at 0%

---

[1] MSc., Center of Excellence Geopolymer and Green Technology, University Malaysia Perlis (UniMAP), 01000, Kangar, Perlis, Malaysia, e-mail: ahmadazrem@unimap.edu.my, ORCID: 0000-0001-5378-3908

[2] PhD., Faculty of Chemical Engineering Technology, University Malaysia Perlis (UniMAP), 01000, Kangar, Perlis, Malaysia, e-mail: mustafa_albakri@unimap.edu.my, ORCID: 0000-0002-9779-8586

[3] PhD., Faculty of Ocean Engineering Technology and Informatics, University Malaysia Terengganu, Terengganu, Malaysia, e-mail: ruzaidi@umt.edu.my, ORCID: 0000-0001-5957-3557

[4] PhD., Faculty of Mechanical Engineering Technology, University Malaysia Perlis (UniMAP), 02600, Arau, Perlis, Malaysia, e-mail: romisuhani@unimap.edu.my, ORCID: 0000-0003-2232-6426

[5] PhD., Faculty of Mechanical Engineering Technology, University Malaysia Perlis (UniMAP), 02600, Arau, Perlis, Malaysia, e-mail: ramadhansyah@ump.edu.my, ORCID: 0000-0002-5255-9856

[6] PhD., Faculty of Mechanical Engineering Technology, University Malaysia Perlis (UniMAP), 02600, Arau, Perlis, Malaysia, e-mail: shayfull@unimap.edu.my, ORCID: 0000-0002-1458-9157

[7] Department of Civil Engineering, Faculty of Engineering – Rabigh Branch, King Abdulaziz University, 21589 Jeddah, Saudi Arabia, e-mail: malmadani@kau.edu.sa, ORCID: 0000-0003-2017-4932

[8] Department of Physics, Częstochowa University of Technology, 42-200, Częstochowa, Poland, e-mail: jerzy.wyslocki@pcz.pl, ORCID: 0000-0002-6157-5065

[9] Prof., Division of Materials Processing Technology and Computer Techniques in Materials Science, Silesian University of Technology, 44-100 Gliwice, Poland, e-mail: agata.sliwa@polsl.pl, ORCID: 0000-0001-9459-3895

[10] Prof., Faculty of Material Science and Engineering, Gheorghe Asachi Technical University of Iasi, 41 D. Mangeron St., 700050 Iasi, Romania, e-mail: sav@tuiasi.ro, ORCID: 0000-0002-9292-749X

(CRGM-0), 5% (CRGM-5), 10% (CRGM-10), 15% (CRGM-15), and 20% (CRGM-20), respectively. NaOH solution (12M) and Na_2SiO_3 solution ratio is set constant at 2.5 for all geopolymer mixture and the fly ash to alkali activator ratio was kept at 2.0. The CRGM at 28 days of curing time was exposed to elevated temperature at 200°C, 400°C, 600°C and 800°C. The weight loss of the CRGM increases with increasing temperature at all elevated temperatures. However, the density and compressive strength of CRGM decrease with an increase of crumb rubber loading for all elevated temperature exposure. The compressive strength of CRGM reduced due to the fact that rubber decomposes between 200°C and 600°C thereby creating voids. CRGM-15 and CRGM-20 showed cracks developed with rough surface at 800°C. Image obtained from scanning electron microscope (SEM) showed that, the CRGM changed significantly due to the decomposition of crumb rubber and evaporation of the free water at 400°C, 600°C and 800°C.

Keywords: fly ash, geopolymer, crumb rubber, elevated temperature exposure

1. Introduction

Geopolymer concrete has been widely researched due to its potential for replacing Ordinary Portland Cement (OPC) as a binder. Fly ash in the geopolymer itself, which is rich with Silicon (Si) and Aluminium (Al) can be activated by an alkaline solution was used as a binder in the concrete mixture instead of cement [1]. The development of fly ash based geopolymer concrete is an attempt to the challenge in producing more environmentally friendly concrete. The use of by-product material such as fly ash as a base material for the concrete binder in replacing OPC through a geopolymerization process has attracted a lot of attention globally [2–7]. In some applications, it is desired that concrete should have low unit weight, high toughness and impact resistance [8,9]. Although concrete is the most commonly used construction material, it does not always fulfil these requirements [10]. Therefore, new applications as rubberized mortar or rubberized concrete have been explored and researched in order to improve elastic properties of concrete as well as to promote recycling waste materials [11–15]. One of the recommended solutions to solve this environmental problem is by incorporating rubber aggregates acquired from unwanted worn tires into the cement concretes [16].

Nowadays, the utilization of recycled rubber in many applications has received a great attention, including its application in constructions [17–20]. In this way, the activity promotes more sustainable and eco-friendly buildings therefore encourages the concept of sustainable production. Rubber aggregate itself has lower density compared to a conventional aggregate thus contribute to the development of semi or fully lightweight concrete. The use of rubber aggregates helps to reach a more economical design [21]. In addition to that, utilization of recycled rubber is proven to be good as fine aggregate for reinforcing concrete as well as several other polymers [9]. Although many studies have been conducted in reinforcing concrete, research focusing on the optimum percentage content of rubber particles incorporated in geopolymer or OPC mortars are still lacking. Previous researchers only highlighted the minimum and maximum contents of rubberized concrete and rubberized concrete mortars, but none have explained on the effects of different composition of it [22]. However, researchers concluded that concrete mortars can be unworkable when the fine natural aggregate replacement by rubber particles exceeds 20% [23–28].

Investigations on the use of fly ash and rubber tire waste in rubberized fly ash based geopolymer concrete are likely to increase, as this offers an environmentally sustainable option of using industrial waste to form useful materials. Geopolymers were reported to possess excellent fire-resistant performance due to their characteristics like ceramic and prepared using alkali activation and alumino-silicate raw materials [29–45]. It was reported that the carbon composite made using geopolymer matrix retains considerable strength even at 1000°C [46].

Kong et al. [36] reported that the strength of fly-ash based geopolymer paste increased 6% after an exposure to elevated temperature of 800°C. While numerous geopolymer systems have been proposed and patented [29], most of them are difficult to work with and require great care in their production. In addition, the polymerization reaction is very sensitive to the temperature and usually requires the geopolymer concrete to be cured at elevated temperature under a strict controlled temperature regime [33, 39]. On the other hand, Luhar [47] investigated on the effects of elevated temperature exposure on rubberized geopolymer concrete and found that, the strength of control and rubberized geopolymer concrete decreased after a thermal exposure up to 600°C. However, compressive strength increased when the exposure to the temperature is greater than 600°C. In addition, it was observed that the compressive strength was found higher at 800°C than that at 600°C [47].

As mortar is a different kind of material which, an increase in the temperature influences both the fine aggregate and the cement paste. Further, the behavior of mortar at high temperatures depends on its composition and the properties of its individual components. The strength of the control and rubberized geopolymer decreases after thermal exposure up to 600°C [47]. Numerous works can be found in the literature on the effects of elevated temperature against normal and high strength concretes [40–48]. Reports showed that concrete exposed to high temperatures loses its compressive strength more than 60% when heated to 800° and probably complete damage when heated above 1000° [36]. Nevertheless, there is a need to investigate rubberized geopolymer mortar when exposed to various elevated temperatures, as the research focusing on this investigation is still lacking. In addition, it is obvious that rubberized geopolymer mortar needs further research in order to clearly understand the behavior of rubber inside the mortar at various temperatures.

2. Raw materials

A low calcium fly ash was used as a source material in the production of crumb rubber geopolymer mortar mixture. This fly ash is classified as a Class F Fly Ash according to the ASTM C618 [49] definitions, due to the relatively low calcium content. The source of the fly ash used was acquired from Sultan Salahuddin Abdul Aziz Power Station, Kapar, Selangor, Malaysia. Crumb Rubber was used in this study in which it was extracted from used tires with maximum size is equivalent to that of fine aggregates such as natural fine aggregates. This crumb rubber is supplied by Gcycle Factory, Sungai Petani, Kedah. The addition of crumb rubber was added in the mixture by weight percentage in steps of 0%, 5%, 10%, 15%, and 20%.

Sodium hydroxide (NaOH) powder with purity 99% (Formosoda-P, Taiwan) was used to produce NaOH solution. Sodium silicate (Na_2SiO_3) was acquired from Musbash Resources Sdn. Bhd., Kuala Lumpur, Malaysia in the form of liquid solution was used in the preparation of alkaline activator. It was in greenish and blue owing color with a density of 2.61 g/cm^3.

3. Mixing method

In order to produce 12 M NaOH solution, 480 g of NaOH powder were dissolved in 1 L distilled water and allowed to cool down at room temperature. The alkaline activator was prepared by mixing NaOH and Na_2SiO_3 and the ratio of Na_2SiO_3 to NaOH was fixed at 2.5. The 2.0 ratio of solid to liquid was kept fixed for all mixtures. The crumb rubber and fly ash were then mixed in a dry state for 30 seconds, then alkaline activator was mixed into the mixtures using a hand mixer for another 10 minutes until a homogenous mixture was formed. Therefore, the different loading of crumb rubber in the fly ash based geopolymer mortar mix was completely prepared.

4. Casting and curing

The geopolymer paste with the addition of crumb rubber was poured into a $50 \times 50 \times 50$ mm steel cube moulds in accordance with ASTM C109 [50] standard and compacted on a vibration table for few minutes. The samples were sealed with a thin plastic film to prevent contaminants and moisture loss. The cube specimens were then left cured at room temperature and demoulded after 24 hours.

After 28 days, cured geopolymers were heated in a Furnace model Carbolite CWF at 200°C, 400°C, 600°C, and 800°C with a heating rate of 4.4°C/min. As soon as the target temperature was attained, the specimens were put inside the furnace for 2 hours. The specimens were then allowed to cool naturally into room temperature inside the furnace. For comparison, one set of the samples was left to cool at ambient temperature (29°C). Three samples were tested for each parameter. Finally, weight loss and compressive strength tests were conducted on the specimens. The chemical changes in specimens after exposure were examined using SEM analysis.

5. Testing

5.1. Compressive strength test

The compressive strength test of crumb rubber geopolymer mortar (CRGM) cubes was performed according to ASTM C109 [50] using a Shimadzu UH-1000kNI Universal

Testing Machine. The force applied on the cube with a loading rate of 1.0 MPa/second and the maximum force was applied to the cube was recorded as compressive strength. Three samples of the cubes were tested on 28 days. The compressive strength of the samples was recorded and analyzed accordingly.

5.2. Density measurement

Density of CRGM was measured in accordance with ASTM C138 [51] standard. Density is one of the physical properties that can be measured by simple procedure and calculated manually with mass per unit volume of samples. Three samples were measured for each parameter. The density measurement of CRGM was conducted after 28 days. The density was calculated using Eq. (5.1) by measuring the dimension and mass of rubberized geopolymer mortar samples.

$$\text{Density (kg/m}^3\text{)} = \frac{M}{V} \tag{5.1}$$

where M is the mass of specimen in kg; and V is the volume of the specimen in m^3.

5.3. Morphology analysis

The SEM was used to carry out the morphology study in accordance with the ASTM B748 [52]. The microstructural of CRGM was observed using JSM-6460 LA model Scanning Electron Microscope (JEOL). The specimens were cut into small pieces and coated with platinum by using Auto Fine Coater, model JEOL JFC 1600 before examination. The microscopic images of the CRGM were captured using SEM on the surface of the mortar cubes after tested under the compression test. The image was then used to analyze the stress distribution and failure mechanism of the CRGM samples after the compression test took place.

6. Result and discussion

6.1. Visual surface appearance analysis

The change of colour and the crack pattern that occur before and after heating in CRGM-0, CRGM-5, CRGM-10, CRGM-15, and CRGM-20 can be seen in Fig. 1 to Fig. 5. The surface crack pattern has been observed on cube mortars after they were exposed to elevated temperatures and cooled to room temperature inside the furnace. Fig. 1 shows the CRGM at room temperature were brownish in colour and had smooth and sharp edges. Fig. 2 shows the colour of the CRGM at 200°C was grey brownish with smooth and sharp edges for all the samples. The change in colour of the mortar's surface is related to the dispersion of carbon black, one of the components of tire rubber particles. Hussin et al. (2015) concluded that almost no cracks developed on the surfaces of samples when heated to 70°C or 200°C [53]. Tang et al. (2021) observed that there are no visible cracks on the surface of the samples after elevated temperature exposure at 200°C. In other words, the appearance of the samples was similar to that at room temperature [54].

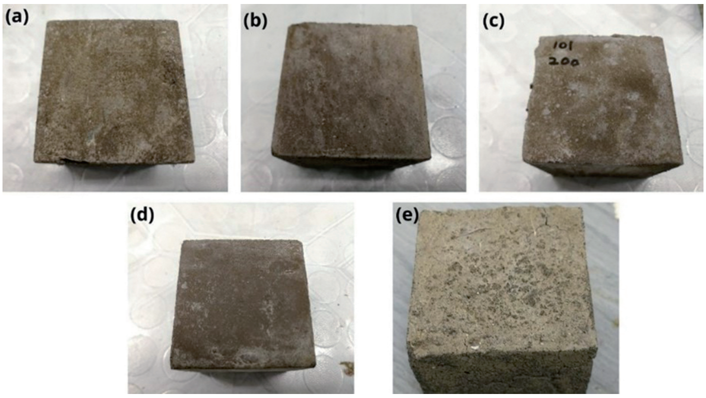

Fig. 1. Visual surface appearance of (a) CRGM-0, (b) CRGM-5, (c) CRGM-10, (d) CRGM-15 and (e) CRGM-20 at room temperature

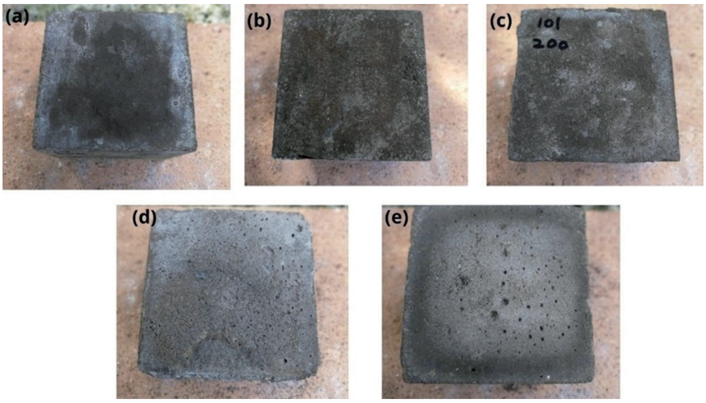

Fig. 2. Visual surface appearance of (a) CRGM-0, (b) CRGM-5, (c) CRGM-10, (d) CRGM-15 and (e) CRGM-20 after temperature exposure at 200°C

At 400°C, the dark grey colour with hairline cracks appeared in CRGM-20, as shown in Fig. 3. The changing in colour was attributed to the burning sign of crumb rubber in the samples. The samples turned brown/black at 400°C due to the dispersion of carbon black, which is a component of rubber tires [55, 56]. Fig. 4 shows that at 600°C, the grey colour with hairline cracks appeared in CRGM-15 and cracks in CRGM-20. At 800°C, all the samples show reddish-brown in colour. The CRGM-10 shows one or two hairline cracks, while CRGM-15 and CRGM-20 show some cracks developed with a rough surface, as seen in Fig. 5. The red colouration is the effect of iron compounds oxidation. Iron is present

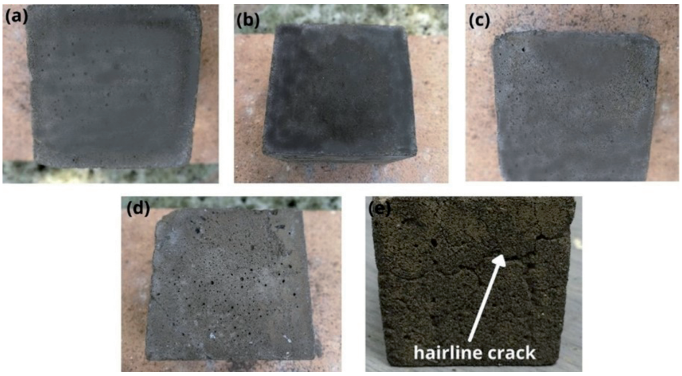

Fig. 3. Visual surface appearance of (a) CRGM-0, (b) CRGM-5, (c) CRGM-10, (d) CRGM-15 and (e) CRGM-20 after temperature exposure at 400°C

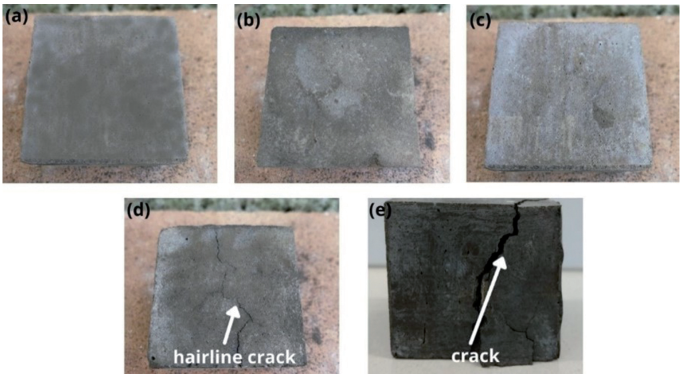

Fig. 4. Visual surface appearance of (a) CRGM-0, (b) CRGM-5, (c) CRGM-10, (d) CRGM-15 and (e) CRGM-20 after temperature exposure at 600°C

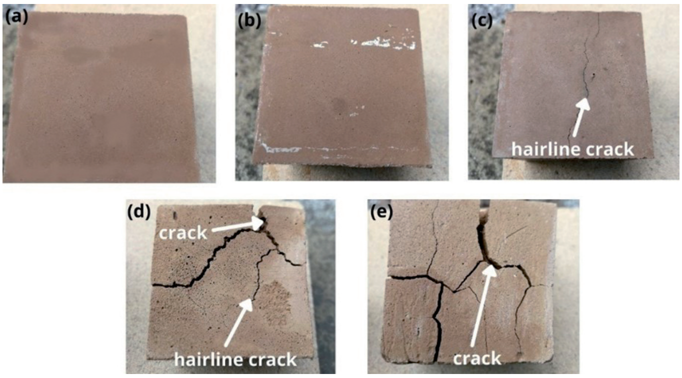

Fig. 5. Visual surface appearance of (a) CRGM-0, (b) CRGM-5, (c) CRGM-10, (d) CRGM-15 and (e) CRGM-20 after temperature exposure at 800°C

mainly in fly ash composition, in oxides with more than 6% of Fe_2O_3 [57]. In addition, at higher temperatures above 600°C, when all crumb rubber was decomposed, the surface colour of these samples changed again and became lighter. Note that the crumb rubber used is a thermoset type that does not melt and just burns. Thus, the produced smoke darkened the samples; however, at the higher temperatures, this soot was eliminated [58].

6.2. Compressive strength analysis

Fig. 6 shows the result of the compressive strength of CRGM at 28 days of curing time. The CRGM were exposed to elevated temperatures at 200°C, 400°C, 600°C, and 800°C, while the control specimen was CRGM at room temperature (RT). The compressive strength of CRGM decreased with the increasing of crumb rubber loading at all elevated temperatures exposure. The compressive strength of CRGM-0, CRGM-5, CRGM-10, CRGM-15, and CRGM-20 at RT were 40.48 MPa, 36.13 MPa, 29.69 MPa, 23.13 MPa, and 20.13 MPa. At 200°C, the CRGM-0 shows compressive strength with 33.94 MPa followed by CRGM-5 (31.47 MPa), CRGM-10 (25.46 MPa), CRGM-15 (22.0 MPa) and CRGM-20 (18.4 MPa). At 400°C, the compressive strength of CRGM-0, CRGM-5, CRGM-10, CRGM-15, and CRGM-20 were 27.9 MPa, 22.87 MPa, 17.96 MPa, 14.13 MPa, and 10.08 MPa. The compressive strength of CRGM-0, CRGM-5, CRGM-10, CRGM-15, and CRGM-20 at 600°C were 23.9 MPa, 20.56 MPa, 15.9 MPa, 13.05 MPa, and 8.76 MPa. In addition, the CRGM-0, CRGM-5, CRGM-10, CRGM-15 and CRGM-20 at 800°C were 20.16 MPa, 18.17 MPa, 14.15 MPa, 11.03 MPa and 7.71 MPa, respectively.

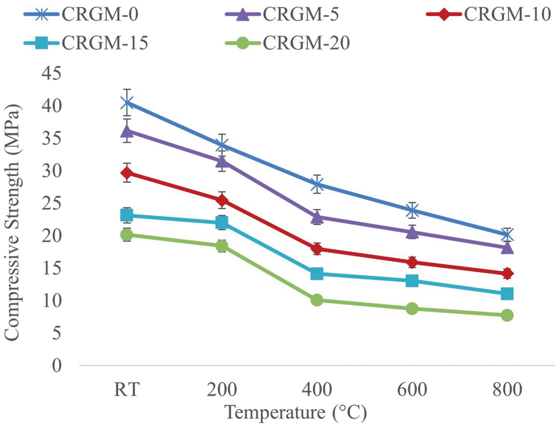

Fig. 6. Compressive strength of CRGM at room temperature and after exposure to elevated temperatures

The compressive strength of the CRGM decreased as the temperature increased from room temperature to 200°C. Compressive strength degradation of the CRGM may be related to the escape of free water from the geopolymer mortar, resulting in internal stresses and ultimately strength degradation. However, CRGM-0 showed a percentage reduction from room temperature to 200°C of 16.16%, slightly higher as compared to CRGM-5, CRGM-10, CRGM-15, and CRGM-20 with 12.90%, 14.25%, 4.89%, and 8.59%. The reasons for a higher strength loss in CRGM-0 during the early stages of temperature exposure are due to the reduction in bonding force and decrease in chain length in Si–O–Al and Si–O–Si regions and due to the expulsion of most of the water present in the geopolymer.

As the temperature increased to reach 400°C, the compressive strength of the CRGM-0 decreased by 17.80% relative to the strength of the corresponding specimen at 200°C. This decrease is due to the start of the evaporation of the chemical water bound in the structure of hydrated geopolymer paste, which is a phenomenon referred to as dehydration. In addition, CRGM-5, CRGM-10, CRGM-15, and CRGM-20 faced a reduction of 27.33%, 29.46%, 35.77%, and 45.22%, respectively, with the main reason attributed to the decomposition of crumb rubber in cube mortars.

However, as the temperature continued to increase from 400°C to 800°C, the compressive strength of CRGM-0 showed a 27.74% reduction. Meanwhile, CRGM-5, CRGM-10, CRGM-15, and CRGM-20 show percentages of strength reduction with 20.55%, 21.21%, 21.94%, and 23.51%, respectively, lower than that of the CRGM-0. This demonstrates that the inclusion of crumb rubber reduces the rate of geopolymer mortar strength loss in general, and the trend is more pronounced at elevated temperatures between 400°C and 800°C. This is mostly due to the crumb rubber when melted at temperatures ranging from 200°C to 400°C, leaving space for water vapour to escape and aiding in the release of pore pressure, hence reducing the mortar's damage.

Luhar et al. (2018) reported that rubberized geopolymer concrete gained strength above 600°C because the amorphous content increased due to the polymerization of unreacted crystalline material, resulting in an increase in the amorphous phase content [47]. This phase content is less in the case of crumb rubber geopolymer concrete due to the decomposition of rubber particles at elevated temperatures, which leads to the formation of voids in the concrete matrix. As explained by Mhaya et al. (2021), this phenomenon is due to the melting of the waste rubber tire crumb (WRTCs), sluggish evaporation of the water, dehydration of the C–A–S–H gels, and decay of $Ca(OH)_2$ that occurred above 400°C in the modified concretes [59]. Guo et al. (2014) and Tang et al. (2021) indicated that the rubber particles melted at high temperatures, which provided more channels to release the water vapor, thus reducing the internal damage and burst caused by the vapor pressure [60, 61].

6.3. Density analysis

Fig. 7 shows the density of CRGM at room temperature and after exposed to elevated temperatures at 200°C, 400°C, 600°C and 800°C. The result indicated that the density of CRGM decreased with increasing of crumb rubber loading for all elevated temperature exposure. The density of CRGM-0 at room temperature gives the highest value of density with 1656 kg/m^3, compared to CRGM-5 (1620 kg/m^3), CRGM-10 (1574 kg/m^3), CRGM-15 (1424 kg/m^3), and CRGM-20 (1378 kg/m^3). At 200°C, the density of CRGM decreased with CRGM-0 (1625 kg/m^3), CRGM-5 (1605 kg/m^3), CRGM-10 (1513 kg/m^3), CRGM-15 (1420 kg/m^3), and CRGM-20 (1357 kg/m^3). As the temperature rising to 400°C, 600°C, and 800°C, the density of CRGM shows a reduction with CRGM-0 (1560, 1472 and 1410 kg/m^3), CRGM-5 (1535, 1427 and 1392 kg/m^3), CRGM-10 (1496, 1404 and 1330 kg/m^3), CRGM-15 (1374, 1368 and 1303 kg/m^3), and CRGM-20 (1348, 1296 and 1288 kg/m^3), respectively.

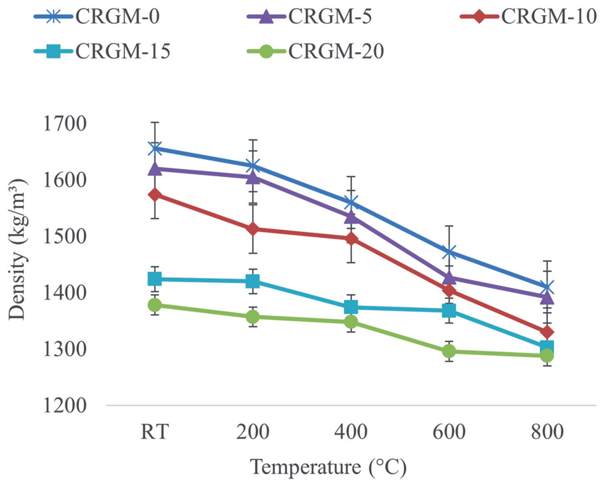

Fig. 7. Density of CRGM at room temperature and after exposed to elevated temperatures

The reduction of density was related to the evaporation of the free water, and chemically bound water in the CRGM evaporate between 20°C to 100°C and between 100°C to 300°C. When the temperature was higher than 300°C, the free water and the chemically bound water inside the CRGM evaporated completely. Sitarz et al. (2018) concluded that the geopolymer mortar decrease in density with the increase of temperature, which results mainly from loss of water [62]. According to Hager et al. (2021), the reduction of density on geopolymer mortar related to the progressive drying process and dihydroxylation. Progressive heating of the geopolymer results in matrix and aggregates drying. Water in the material is gradually removed from the heated matrix. With temperature increase, free water is removed first, then adsorbed water evaporates. At higher temperature, the water bound physically in the form of hydroxyl groups is removed [57]. According to Duxson et al. (2007), the dihydroxylation process that takes place at 200°C to 600°C induces shrinkage of the geopolymer binder. Nevertheless, the geopolymer binder may experience an expansion attributed to the thermal dilation of water around 120°C. As soon as the drying process starts, intensive shrinkage begins [63].

6.4. Weight loss analysis

Fig. 8 represents the weight loss of CRGM samples exposed to elevated temperatures. From the results, the weight loss of the CRGM increased with increasing temperature at all elevated temperatures, respectively. The weight loss of CRGM at elevated temperatures exposure shows an increment with CRGM-0 (5.16%, 14.22%, 16.43%, and 18.69%), CRGM-5 (5.35%, 15.67%, 17.33%, and 19.67%), CRGM-10 (5.5%, 18.75%, 19.3%, and 21.91%), CRGM-15 (5.98%, 19.23%, 21.1%, and 23.44%) and CRGM-20 (6.44%, 20.1%, 23.7%, and 25.63%).

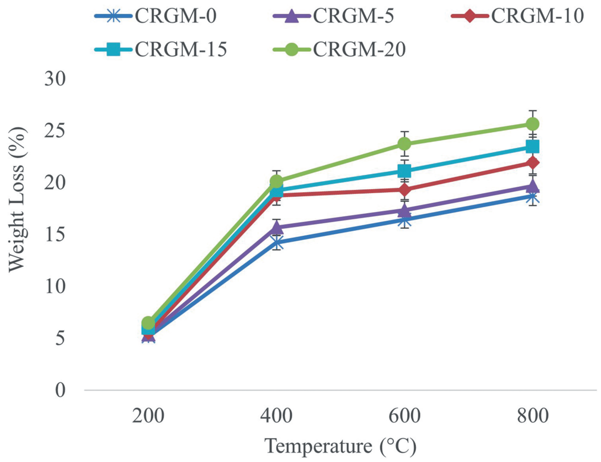

Fig. 8. Weight loss of CRGM after exposure to elevated temperatures

During the heating process from room temperature to 200°C, the weight loss of CRGM occurred due to the evaporation of both the free water and part of the chemically bonded water from the geopolymer. Under 100°C, the physically bonded water or free water located in the pores evaporates, which contributes about 5.5% to 6.5% of the total water content in the geopolymer mortar. Then, further heating above 100°C leads to evaporation of the chemically bonded water and the hydroxyl group OH inside the gel pores. Consequently, pore pressure grows gradually in the pore structures as a result of heat transfer and moisture evaporation. When the vapor pressure comes to the maximum limit of the matrix, intensive thermal cracking and spalling occur. Generally, two effects are believed to be responsible for the mass loss of geopolymers at high temperatures, which are water evaporation and dihydroxylation. Hardened geopolymer materials contain physical water, chemical water, and hydroxyl. Each type of water evaporates at a different temperature range; physical water and chemical water evaporate at 20°C to 100°C and 100°C to 300°C, respectively, while hydroxyl groups evaporate at temperatures above 300°C [64].

At 200°C to 400°C, there was a sudden increment in the percentage of weight loss of the CRGM by 63% to 68%. This is due to evaporation of the chemically bonded water in the structure and the decomposition of crumb rubber of geopolymer mortar. AbdulKadir (2018) reported that this little change is because the elevated temperature (200°C) could evaporate the water entrapped in the mortar. Still, it was insufficient to evaporate the rubber in the samples as the authors tested its evaporation temperature to be 260°C. As the temperature rose above 300°C, more crumb rubber melted, forming more void networks that became points of crack propagation when a load was applied, resulting in a dramatic decrease in weight loss and strength from 200°C to 400°C [65]. Meanwhile, according to Wu and Li (2017), there are two reasons for this type of behavior. The first reason is that the crumb rubber may have melted at a moderate temperature of 200°C to 300°C, resulting in the formation of interconnected channels to relieve internal pressure. The second mechanism is that when a load is applied to these interconnected void channels, they become points of weakness [66]. In addition, when the temperature reaches above 300°C, the dihydroxylation of OH groups happen with the subsequent polycondensation into the siloxo bond Si–O–Si, linking neighbouring geopolymeric micelles. It is proven that more than 70% of the reaction water is physically bonded water, which evaporates before 100°C without causing any internal stress and remarkable shrinkage. However, many micropores will be produced owing to the empty space left by the water evaporation. The remaining 30% water, chemically bonded water, and hydroxyl groups OH, account for up to 90% of the total shrinkage when the samples are heated from 20°C to 500°C [67].

On the other hand, the weight loss percentage of CRGM increased at 400°C and continued to 800°C. As can be seen in Figure 8, the weight loss percentage of CRGM reached its peak when the samples were heated to 800°C. This is related to the decomposition of carbonate species such as sodium and calcium carbonates. As reported by He et al. (2020), at higher temperatures, the weight loss of geopolymer is attributed to a dehydration process of the structure and the decomposition of the carbonates present [64]. Fawzy et al. (2020) concluded that the mass loss above 400°C is mainly because of the decomposition of calcium silicate hydrate (C–S–H) as mortar includes capillary water and physically absorbed water (gel water) and chemically bonded water in C–S–H and ($Ca(OH)_2$). Capillary water and physically absorbed water take up a large amount of cement-paste weight and can be released from mortar by evaporation when the temperature is about 200°C or above. On the other hand, chemically bonded water is a part of cement hydrate compound sand called non-evaporable water because it cannot be removed from cement paste before the decomposition of the C–S–H at high temperatures [68]. In addition, Mahmod et al. (2017) concluded that this behavior is due to the elimination of free water from the capillary pores by evaporation in addition to the loss of chemically bound water [69]. According to Aslani & Asif (2019), when geopolymer concrete is exposed to elevated temperatures, it can lose its stiffness and mechanical properties. Increased temperature and weight loss cause the structural integrity of geopolymer concrete to deteriorate. The decomposing of the crumb rubber also reduced the weight loss of the self-compacting rubberized concrete (SCRC). This creates pores within the matrix, reducing weight loss by generating internal stresses when the load is applied. As the amount of crumb rubber increases, so does the number of such voids, resulting in even more weight loss and strength reduction [70].

6.5. Morphology analysis

The SEM image analysis was carried out on CRGM taken from their respective mortar samples. Fig. 9 to Fig. 12 show the SEM images of CRGM after being exposed to 200°C, 400°C, 600°C, and 800°C. As shown in Fig. 9, the SEM image of CRGM exposed to 200°C shows that the free water evaporated completely, and the crumb rubber also did not decompose at this temperature exposure. Fig. 10 shows the SEM image of the CRGM changed significantly due to the decomposition of crumb rubber at 400°C. This decomposition will lead to pores appearing, which can release the vapor pressure inside the CRGM. As reported by Gupta et al. (2017), rubber fibres were decomposed at higher temperatures and longer exposure duration (300°C and 120 min exposure duration), which created voids in concrete [71].

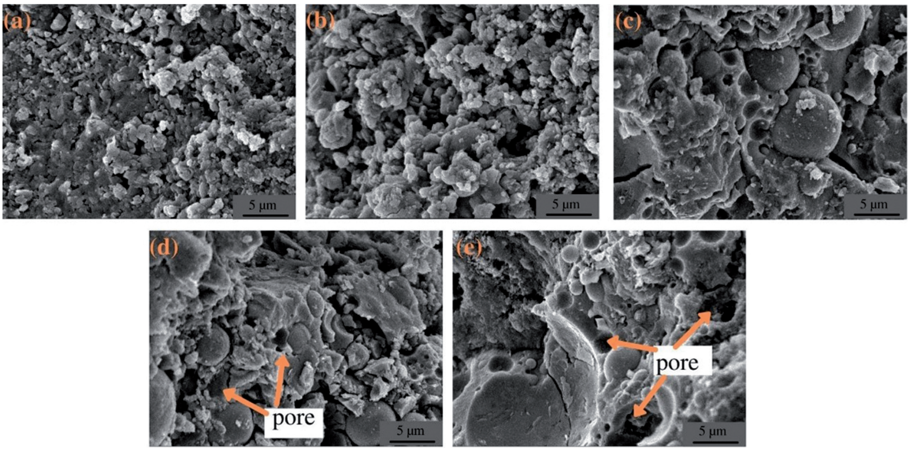

Fig. 9. SEM images of (a) CRGM-0, (b) CRGM-5, (c) CRGM-10, (d) CRGM-15 and (e) CRGM-20 after exposure to 200°C at 3000$^\times$ magnification

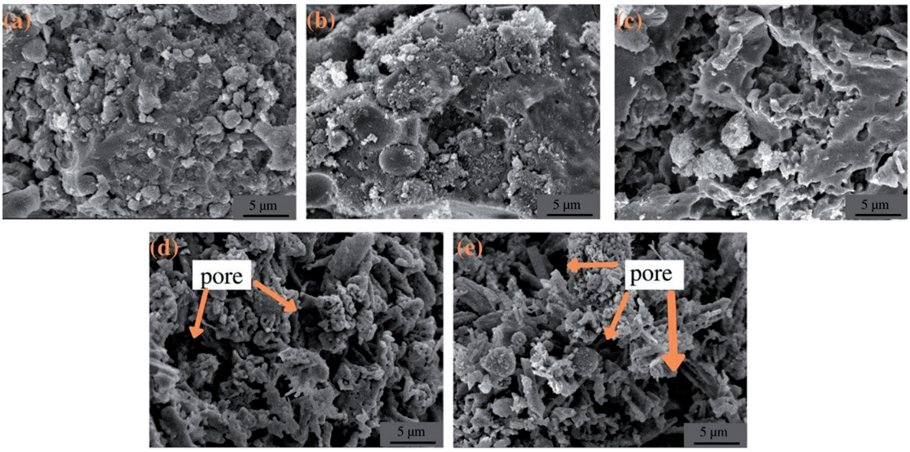

Fig. 10. SEM images of (a) CRGM-0, (b) CRGM-5, (c) CRGM-10, (d) CRGM-15 and (e) CRGM-20 after exposure to 400°C at 3000$^\times$ magnification

Fig. 11 and Fig. 12 show that almost all the crumb rubber decomposed and free water inside the CRGM evaporated completely after heating to 600°C and 800°C, leaving only some residues and pores. This resulted in additional crack development and deterioration of the internal structure of the geopolymer mortar. As shown in Fig. 12, the decomposition of most of the $Ca(OH)_2$ resulted in some broken phases with no connected crystals and a loose internal structure. Therefore, the macro-cracks and mass loss of the specimens increased while the internal structure density decreased, leading to a significant decrease in the mechanical properties. The hydrate change in the geopolymer mortar was obvious, indicating a decline in the integrity of the samples. The mechanism of CRGM at elevated temperature exposure at 200°C, 400°C, 600°C, and 800°C is shown in Fig. 13.

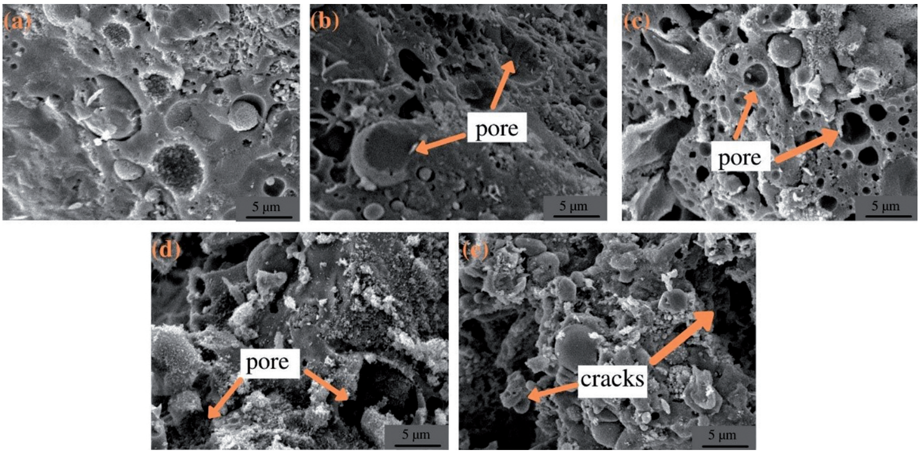

Fig. 11. SEM images of (a) CRGM-0, (b) CRGM-5, (c) CRGM-10, (d) CRGM-15 and (e) CRGM-20 after exposure to 600°C at $3000^{\times}$ magnification

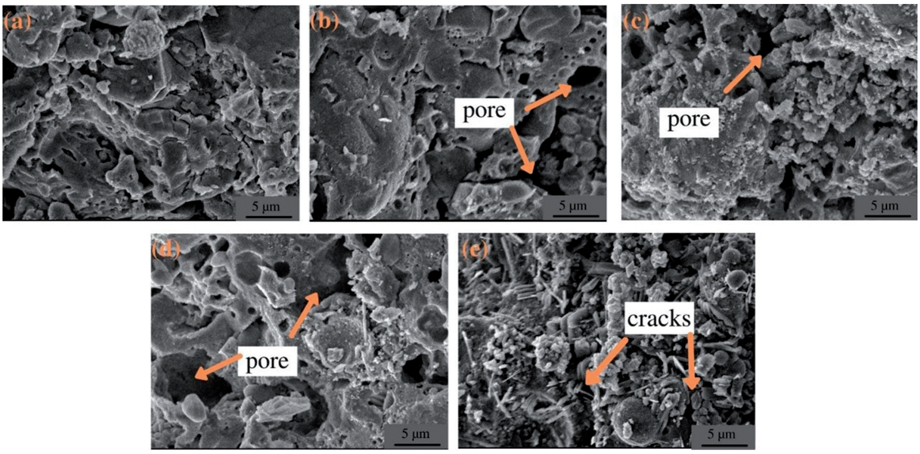

Fig. 12. SEM images of (a) CRGM-0, (b) CRGM-5, (c) CRGM-10, (d) CRGM-15 and (e) CRGM-20 after exposure to 800°C at $3000^{\times}$ magnification

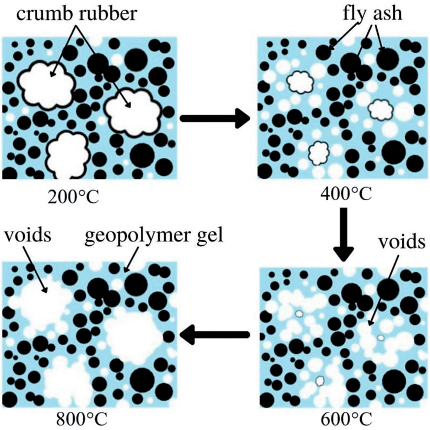

Fig. 13. The mechanism of CRGM at elevated temperature exposure at 200°C, 400°C, 600°C, and 800°C

7. Conclusions

It can be concluded that, the fine aggregate in geopolymer mortar is replaceable by crumb rubber loading until 20%. The different percentage composition of rubber crumb in the CRGM highly influence the results of the compressive strength, density, water absorption capacity and SEM micrograph. CRGM exposed to elevated temperatures at 200°C, 400°C, 600°C, and 800°C show a reduction in compressive strength and density while the weight loss increases with an increase in the amount of crumb rubber. The micrograph of CRGM changes significantly due to the decomposition of crumb rubber and evaporation of the free water at 400°C. This decomposition leads to appearance of pores releasing the vapor pressure inside the RGM, therefore decreases in mechanical properties.

Acknowledgements

Special thanks to Center of Excellence Geopolymer and Green Technology (CEGeoGTech) and Faculty of Chemical Engineering Technology, Universiti Malaysia Perlis (UniMAP) for providing research facilities and technical assistance for their help and cooperation in research work. The authors would like to extend their gratitude to Department of Physics, Częstochowa University of Technology, Częstochowa, Poland.

References

[1] D. Hardjito, B.V. Rangan, *Development and properties of low-calcium fly ash-based geopolymer concrete*. Curtin University of Technology, Curtin Publishing, 2005.

[2] M.M.A.B. Abdullah, K. Hussin, M. Bnhussain, et al., "Fly ash-based geopolymer lightweight concrete using foaming agent", *International Journal of Molecular Sciences*, 2012, vol. 13, no. 6, pp. 7186–7198, DOI: 10.3390/ijms13067186.

[3] F. Pacheco-Torgal, A. Shasavandi, S. Jalali, "Eco-efficient concrete using industrial wastes: A review", *Materials Science Forum*, 2012, vol. 730–732, pp. 581–586, DOI: 10.4028/www.scientific.net/msf.730-732.581.

[4] A. Mohajerani, D. Suter, T. Jeffrey-Bailey, et al., "Recycling waste materials in geopolymer concrete", *Clean Technologies and Environmental Policy*, 2019, vol. 21, no. 3, pp. 493–515, DOI: 10.1007/s10098-018-01660-2.

[5] O.H. Li, et al., "Evaluation of the effect of silica fume on amorphous fly ash geopolymers exposed to elevated temperature", *Magnetochemistry*, 2021, vol. 7, no. 1, DOI: 10.3390/magnetochemistry7010009.

[6] W.W.A. Zailani, et al., "Characterisation at the Bonding Zone between Fly Ash Based Geopolymer Repair Materials (GRM) and Ordinary Portland Cement Concrete (OPCC)", *Materials*, 2021, vol. 14, no. 1, DOI: 10.3390/ma14010056.

[7] N.F. Shahedan, et al., "Properties of a New Insulation Material Glass Bubble in Geopolymer Concrete", *Materials*, 2021, vol. 14, no. 4, DOI: 10.3390/ma14040809.

[8] T. Edeska¨r, *Technical and environmental properties of tyre shreds focusing on ground engineering applications*. Lulea°, 2004.

[9] Y. Grohens, S.K. Kumar, A. Boudenne, Y. Weimin, Eds., *Recycling and reuse of materials and their products*. CRC Press, 2013.

[10] M. Chemrouk, "The Deteriorations of Reinforced Concrete and the Option of High Performances Reinforced Concrete", *Procedia Engineering*, 2015, vol. 125, pp. 713–724, DOI: 10.1016/j.proeng.2015.11.112.

[11] I.B. Topçu, "The properties of rubberized concretes", *Cement and Concrete Research*, 1995, vol. 25, no. 2, pp. 304–310, DOI: 10.1016/0008-8846(95)00014-3.

[12] İ.B. Topçu, "Assessment of the brittleness index of rubberized concretes", *Cement and Concrete Research*, 1997, vol. 27, no. 2, pp. 177–183, DOI: 10.1016/s0008-8846(96)00199-8.

[13] M. Emiroglu, S. Yildiz, O. Keleştemur, M.H. Keleştemur, "Bond performance of rubber particles in the self-compacting concrete", *Bond in Concrete*. 2012, pp. 779–786.

[14] Y. El-Sherbini, A.K. Abdel-Gawad, A. Shalaby, A. El-Gammal, "Compressive strength of concrete utilizing waste tire rubber", *Journal of Emerging Trends in Engineering and Applied Sciences*, 2010, vol. 1, no. 1, pp. 96–99.

[15] B.D. Reddy, S.A. Jyothy, P.R. Babu, "Experimental investigation on concrete by partially replacement of ware aggregate with junk rubber", *The International Journal of Engineering and Science*, 2013, vol. 2, no. 12, pp. 61–65.

[16] Z. Boudaoud, M. Beddar, "Effects of Recycled Tires Rubber Aggregates on the Characteristics of Cement Concrete", *Open Journal of Civil Engineering*, 2012, vol. 2, no. 4, pp. 193–197, DOI: 10.4236/ojce.2012.24025.

[17] D. Raghavan, H. Huynh, C.F. Ferraris, "Workability, mechanical properties, and chemical stability of a recycled tyre rubber-filled cementitious composite", *Journal of Materials Science*, 1998, vol. 33, no. 7, pp. 1745–1752, DOI: 10.1023/a:1004372414475.

[18] F. Hernández-Olivares, G. Barluenga, M. Bollati, B. Witoszek, "Static and dynamic behaviour of recycled tyre rubber-filled concrete", *Cement and Concrete Research*, 2002, vol. 32, no. 10, pp. 1587–1596, DOI: 10.1016/s0008-8846(02)00833-5.

[19] K. Kovler, N. Roussel, "Properties of fresh and hardened concrete," *Cement and Concrete Research*, 2011, vol. 41, no. 7, pp. 775–792, DOI: 10.1016/j.cemconres.2011.03.009.

[20] İ.B. Topçu, T. Bilir, "Experimental investigation of some fresh and hardened properties of rubberized self-compacting concrete", *Materials & Design*, 2009, vol. 30, no. 8, pp. 3056–3065, DOI: 10.1016/j.matdes.2008.12.011.

[21] M.K. Ismail, A.A.A. Hassan, "Performance of Full-Scale Self-Consolidating Rubberized Concrete Beams in Flexure", *ACI Materials Journal*, 2016, vol. 113, no. 2, pp. 207–218.

[22] I.B. Topçu, T. Uygunoglu, "Sustainability of using waste rubber in concrete", in *Sustainability of Construction Materials*. Woodhead Publishing, 2016, pp. 597–623.

[23] A.C. Ho, A. Turatsinze, R. Hameed, D.C. Vu, "Effects of rubber aggregates from grinded used tyres on the concrete resistance to cracking", *Journal of Cleaner Production*, 2012, vol. 23, no. 1, pp. 209–215, DOI: 10.1016/j.jclepro.2011.09.016.

[24] N. Deshpande, S.S. Kulkarni, T. Pawar, V. Gunde, "Experimental investigation on strength characteristics of concrete using tyre rubber as aggregates in concrete", *International Journal of Applied Engineering Research and Development*, 2014, vol. 4, no. 2, pp. 97–108.

[25] R. Hooton, M. Nehdi, A. Khan, "Cementitious Composites Containing Recycled Tire Rubber: An Overview of Engineering Properties and Potential Applications", *Cement, Concrete and Aggregates*, 2001, vol. 23, no. 1, DOI: 10.1520/cca10519j.

[26] T.-H. Nguyen, A. Toumi, A. Turatsinze, F. Tazi, "Restrained shrinkage cracking in steel fibre reinforced and rubberised cement-based mortars", *Materials and Structures*, 2011, vol. 45, no. 6, pp. 899–904, DOI: 10.1617/s11527-011-9806-x.

[27] M.M.A.B. Abdullah, S.N.F.S. Adam, M.A. Bakar, K. Leong, "Comparison of rubber as aggregate and rubber as filler in concrete", in *Proceedings of the 1st International Conference on Sustainable Materials 2007 (ICoMS 2007), Penang, Malaysia.* 2007.

[28] N.A. Emira, N.S. Bajaba, "The effect of rubber crumbs addition on some mechanical properties of concrete composites", *International Journal of Mechanic Systems Engineering*, 2012, vol. 2, no. 2, pp. 53–58.

[29] J. Davidovits, "30 years of successes and failures in geopolymer applications-Market trends and potential breakthroughs", in *Proceedings of the Geopolymer 2002 Conference, October 28-29, Melbourne, Australia*. 2002.

[30] T.W. Cheng, J.P. Chiu, "Fire-resistant geopolymer produced by granulated blast furnace slag", *Minerals Engineering*, 2003, vol. 16, no. 3, pp. 205–210, DOI: 10.1016/s0892-6875(03)00008-6.

[31] V.F.F. Barbosa, K.J.D. MacKenzie, "Thermal behaviour of inorganic geopolymers and composites derived from sodium polysialate", *Materials Research Bulletin*, 2003, vol. 38, no. 2, pp. 319–331, DOI: 10.1016/s0025-5408(02)01022-x.

[32] V.F.F. Barbosa, K.J.D. MacKenzie, "Synthesis and thermal behaviour of potassium sialate geopolymers", *Materials Letters*, 2003, vol. 57, no. 9–10, pp. 1477–1482, DOI: 10.1016/s0167-577x(02)01009-1.

[33] D. Hardjito, S.E. Wallah, D.M. Sumajouw, B.V. Rangan, "On the Development of Fly Ash-Based Geopolymer Concrete", *ACI Materials Journal*, 2004, vol. 101, no. 6, pp. 467–472, DOI: 10.14359/13485.

[34] T. Bakharev, "Geopolymeric materials prepared using Class F fly ash and elevated temperature curing", *Cement and Concrete Research*, 2005, vol. 35, no. 6, pp. 1224–1232, DOI: 10.1016/j.cemconres.2004.06.031.

[35] T. Bakharev, "Resistance of geopolymer materials to acid attack", *Cement and Concrete Research*, 2005, vol. 35, no. 4, pp. 658–670, DOI: 10.1016/j.cemconres.2004.06.005.

[36] D.L.Y. Kong, J.G. Sanjayan, K. Sagoe-Crentsil, "Comparative performance of geopolymers made with metakaolin and fly ash after exposure to elevated temperatures", *Cement and Concrete Research*, 2007, vol. 37, no. 12, pp. 1583–1589, DOI: 10.1016/j.cemconres.2007.08.021.

[37] D.L.Y. Kong, J.G. Sanjayan, "Damage behavior of geopolymer composites exposed to elevated temperatures", *Cement and Concrete Composites*, 2008, vol. 30, no. 10, pp. 986–991, DOI: 10.1016/j.cemconcomp.2008.08.001.

[38] D.L.Y. Kong, J.G. Sanjayan, "Effect of elevated temperatures on geopolymer paste, mortar and concrete", *Cement and Concrete Research*, 2010, vol. 40, no. 2, pp. 334–339, DOI: 10.1016/j.cemconres.2009.10.017.

[39] N. Lloyd, V. Rangan, "Geopolymer Concrete—Sustainable Cementless Concrete", in *10th ACI International Conference on Recent Advances in Concrete Technology and Sustainability Issues*. Farmington Hills, MI: American Concrete Institute, 2009.

[40] O. Abdulkareem, M. Abdullah, K. Hussin, et al., "Mechanical and Microstructural Evaluations of Lightweight Aggregate Geopolymer Concrete before and after Exposed to Elevated Temperatures", *Materials*, 2013, vol. 6, no. 10, pp. 4450–4461, DOI: 10.3390/ma6104450.

[41] O.A. Abdulkareem, A.M.M. Al Bakri, H. Kamarudin, et al., "Effects of elevated temperatures on the thermal behavior and mechanical performance of fly ash geopolymer paste, mortar and lightweight concrete", *Construction and Building Materials*, 2014, vol. 50, pp. 377–387, DOI: 10.1016/j.conbuildmat.2013.09.047.

[42] O.A. Abdulkareem, A.M.M. Al Bakri, H. Kamarudin, et al., "Fire Resistance Evaluation of Lightweight Geopolymer Concrete System Exposed to Elevated Temperatures of 100–800°C", *Key Engineering Materials*, 2013, vol. 594–595, pp. 427–432, DOI: 10.4028/www.scientific.net/kem.594-595.427.

[43] H.C. Yong, L.Y. Ming, A.M.M. Al Bakri, et al., "Fire Resistant Properties of Geopolymers: A Review", *Key Engineering Materials*, 2015, vol. 660, pp. 39–43, DOI: 10.4028/www.scientific.net/kem.660.39.

[44] H.C. Yong, L.Y. Ming, A.M.M. Al Bakri, K. Hussin, "Thermal Resistance Variations of Fly Ash Geopolymers: Foaming Responses", *Scientific Reports*, 2017, vol. 7, no. 1, DOI: 10.1038/srep45355.

[45] N. Ariffin, et al., "Effect of Aluminium Powder on Kaolin-Based Geopolymer Characteristic and Removal of Cu2+", *Materials*, 2021, vol. 14, no. 4, DOI: 10.3390/ma14040814.

[46] A. Foden, R. Lyon, P. Balaguru, et al., "High temperature inorganic resin for use in fiber reinforced composites", in *Proceedings of 1st international conference on composites in infrastructures*, University of Arizona. 1996, pp. 166–177.

[47] S. Luhar, S. Chaudhary, I. Luhar, "Thermal resistance of fly ash based rubberized geopolymer concrete", *Journal of Building Engineering*, 2018, vol. 19, pp. 420–428, DOI: 10.1016/j.jobe.2018.05.025.

[48] A. Hassan, M. Arif, M. Shariq, "Mechanical Behaviour and Microstructural Investigation of Geopolymer Concrete After Exposure to Elevated Temperatures", *Arabian Journal for Science and Engineering*, 2020, vol. 45, no. 5, pp. 3843–3861, DOI: 10.1007/s13369-019-04269-9.

[49] *ASTM C168 Standard terminology relating to thermal insulation*. ASTM International, West Conshohocken, PA, 2017.

[50] *ASTM C109 Standard Test Method for Compressize Strength of Hydraulic Cement Mortar*. ASTM International, West Conshohocken, PA, 2020.

[51] *ASTM C138 Standard Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete*. ASTM International, West Conshohocken, PA, 2017.

[52] *ASTM B748 Standard Test Method for Measurement of Thickness of Metallic Coatings by Measurement of Cross Section with a Scanning Electron Microscope*. ASTM International, West Conshohocken, PA, 2016.

[53] M.W. Hussin, M.A.R. Bhutta, M. Azreen, et al., "Performance of blended ash geopolymer concrete at elevated temperatures", *Materials and Structures*, 2015, vol. 48, no. 3, pp. 709–720, DOI: 10.1617/s11527-014-0251-5.

[54] Y. Tang, W. Feng, Z. Chen, et al., "Fracture behavior of a sustainable material: Recycled concrete with waste crumb rubber subjected to elevated temperatures", *Journal of Cleaner Production*, 2021, vol. 318, art. ID 128553, DOI: 10.1016/j.jclepro.2021.128553.

[55] A.M. Marques, J.R. Correia, J. de Brito, "Post-fire residual mechanical properties of concrete made with recycled rubber aggregate", *Fire Safety Journal*, 2013, vol. 58, pp. 49–57, DOI: 10.1016/j.firesaf.2013.02.002.

[56] M.A. Muhammad, W.A.A. Faraj, M.R. Abdul-Kadir, "Effect of Impact Load on Concrete Containing Recycled Tire Rubber Aggregate with and without Fire Exposure", *Kurdistan Journal of Applied Research*, 2020, vol. 5, no. 1, pp. 49–65, DOI: 10.24017/science.2020.1.4.

[57] I. Hager, M. Sitarz, K. Mróz, "Fly-ash based geopolymer mortar for high-temperature application – Effect of slag addition", *Journal of Cleaner Production*, 2021, vol. 316, art. ID 128168, DOI: 10.1016/j.jclepro.2021.128168.

[58] M. Mousavimehr, M. Nematzadeh, "Predicting post-fire behavior of crumb rubber aggregate concrete", *Construction and Building Materials*, 2019, vol. 229, art. ID 116834, DOI: 10.1016/j.conbuildmat.2019.116834.

[59] A.M. Mhaya, M.H. Baghban, I. Faridmehr, et al., "Performance Evaluation of Modified Rubberized Concrete Exposed to Aggressive Environments", *Materials*, 2021, vol. 14, no. 8, art. ID 1900, DOI: 10.3390/ma14081900.

[60] Y. Guo, J. Zhang, G. Chen, Z. Xie, "Compressive behaviour of concrete structures incorporating recycled concrete aggregates, rubber crumb and reinforced with steel fibre, subjected to elevated temperatures", *Journal of Cleaner Production*, 2014, vol. 72, pp. 193–203, DOI: 10.1016/j.jclepro.2014.02.036.

[61] Y. Tang, W. Feng, Z. Chen, et al., "Experimental and Theoretical Investigation on the Thermo-Mechanical Properties of Recycled Aggregate Concrete Containing Recycled Rubber", *Frontiers in Materials*, 2021, vol. 8, DOI: 10.3389/fmats.2021.655097.

[62] M. Sitarz, I. Hager, J. Kochanek, "Effect of High Temperature on Mechanical Properties of Geopolymer Mortar", *MATEC Web of Conferences*, 2018, vol. 163, art. ID 06004, DOI: 10.1051/matecconf/201816306004.

[63] P. Duxson, G.C. Lukey, J.S.J. van Deventer, "Physical evolution of Na-geopolymer derived from metakaolin up to 1000°C", *Journal of Materials Science*, 2007, vol. 42, no. 9, pp. 3044–3054, DOI: 10.1007/s10853-006-0535-4.

[64] J. He, G. Zhang, "Geopolymerization of red mud and fly ash for civil infrastructure applications", in *Geo-Frontiers 2011: Advances in Geotechnical Engineering,* 2011, pp. 1287–1296.

[65] M.R. AbdulKadir, et al., "Effect of High Temperature on Mechanical Properties of Rubberized Concrete Using Recycled Tire Rubber as Fine Aggregate Replacement", *Engineering and Technology Journal*, 2018, vol. 36, no. 8A, DOI: 10.30684/etj.36.8a.10.

[66] C. Wu, V.C. Li, "Thermal-mechanical behaviors of CFRP-ECC hybrid under elevated temperatures", *Composites Part B: Engineering*, 2017, vol. 110, pp. 255–266, DOI: 10.1016/j.compositesb.2016.11.037.

[67] J. Davidovits, *Geopolymer Chemistry And Applications*, 5th ed. Geopolymer Institute, 2020.

[68] H. Fawzy, S. Mustafa, A. Abd El Badie, "Effect of Elevated Temperature on Concrete Containing Waste Tires Rubber", *Egyptian Journal for Engineering Sciences and Technology*, 2020, vol. 29, no. 1, pp. 1–13, DOI: 10.21608/eijest.2020.97315.

[69] H.M. Mahmod, A.A.F.N. Aznieta, S.J. Gatea, "Evaluation of rubberized fibre mortar exposed to elevated temperature using destructive and non-destructive testing", *KSCE Journal of Civil Engineering*, 2017, vol. 21, no. 4, pp. 1347–1358, DOI: 10.1007/s12205-016-0721-0.

[70] F. Aslani, Z. Asif, "Properties of Ambient-Cured Normal and Heavyweight Geopolymer Concrete Exposed to High Temperatures", *Materials*, 2019, vol. 12, no. 5, DOI: 10.3390/ma12050740.

[71] T. Gupta, S. Siddique, R.K. Sharma, S. Chaudhary, "Effect of elevated temperature and cooling regimes on mechanical and durability properties of concrete containing waste rubber fiber", *Construction and Building Materials*, 2017, vol. 137, pp. 35–45, DOI: 10.1016/j.conbuildmat.2017.01.065.

Received: 20.09.2022, Revised: 22.03.2022