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FELLIPE RODRIGUES ANDRÉ

**PERFORMANCE OF CEMENTITIOUS COMPOSITES USING
MODIFIED WASTE TIRE RUBBER AS RECYCLED AGGREGATE**

SÃO PAULO – SP

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Thesis submitted to Stricto Sensu Graduate Program in Civil Engineering, São Judas Tadeu University in Partial Fulfillment of The Requirements for The Degree of Master of Science in Civil Engineering.

Advisor: Prof. Dr. Mostafa Galal Abdelfattah Mohamed Aboelkheir

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ABSTRACT

The tire rubber, although widely consumed in the automotive industry, creates a challenge to sustainable development due to its non-degradable nature and recycling difficulty attributed to vulcanization. In this regard, the academic world seeks viable alternatives for reutilizing elastomers through various treatments. For over 25 years, the exploration of this polymer as an aggregate in cementitious composites has been a subject of research. However, the reduction of the mechanical properties arises from the weak interface between cement paste and rubber, due to the hydrophobic nature of the polymer, increased by the vulcanization process. This work aims to prepare mortar floor boards by volumetrically replacing fine aggregate with untreated and chemically treated ground tire rubber (GTR). A Vinyl Acetate emulsion was also tested as an admixture additive. At first, two treatment reagents were tested: hydrogen peroxide and potassium permanganate during three intervals of time; 1h, 2h, and 3h. Consequently, the GTR samples were characterized by contact angle, crosslinking degree, and Fourier-transform infrared spectroscopy (FTIR), highlighting the potassium permanganate for 3 hours as the optimal treatment. Prismatic specimens were cast for water absorption, voids ratio, and bulk density tests, alongside mechanical tests for compressive and flexural strength, and dynamic modulus of elasticity. The monitoring of the thermal behavior of the rubberized floor boards was through the use of thermocouples during the exposure to solar radiation in an open environment. The volumetric replacement of 10% of fine aggregate by the treated GTR showed that the results did not undergo a significant change closest to control samples for all conducted tests. The treated GTR caused 21% increase in compressive strength and 27% in flexural strength. The disposal of waste tires for use as recycled aggregate in cementitious composites reduces the inappropriate disposal of the product, contributing to sustainable development in its 3 pillars, environmental, social and economic, and also contributes to the Sustainable Development Goals (SDG) 3, 6, 11, 12, 13 and 15 of the 2030 Agenda.

Keywords: rubberized mortar; recycled aggregate; end of life tire; chemical treatment, agenda 2030; sustainability.

RESUMO

A borracha de pneu, embora amplamente consumida na indústria automotiva, apresenta um desafio para o desenvolvimento sustentável devido à sua natureza não degradável e à dificuldade de reciclagem atribuída à vulcanização. Nesse sentido, o meio acadêmico busca alternativas viáveis de reaproveitamento de elastômeros por meio de diversos tratamentos. Há mais de 25 anos, a exploração desse polímero como agregado em compósitos cimentícios tem sido objeto de pesquisa. Entretanto, a redução das propriedades mecânicas decorre da fraca interface entre a pasta de cimento e a borracha, devido à natureza hidrofóbica do polímero, potencializada pelo processo de vulcanização. Este trabalho tem como objetivo a preparação de placas de argamassa para pisos, substituindo volumetricamente o agregado fino por borracha de pneu moída (GTR) não tratada e tratada quimicamente. Uma emulsão de Acetato de Vinila também foi testada como aditivo de mistura. Inicialmente, foram testados dois agentes de tratamento: peróxido de hidrogênio e permanganato de potássio em três intervalos de tempo; 1h, 2h e 3h. Consequentemente, as amostras GTR foram caracterizadas por ângulo de contato, grau de reticulação e espectroscopia de infravermelho por transformada de Fourier (FTIR), destacando o uso do permanganato de potássio por 3 horas como o melhor tratamento aplicado. Corpos de prova prismáticos foram moldados para os ensaios de absorção de água, índice de vazios e densidade aparente, juntamente com testes mecânicos de resistência à compressão e flexão e módulo dinâmico de elasticidade. O monitoramento do comportamento térmico das placas de piso emborrachadas foi através do uso de termopares durante exposição à radiação solar em ambiente aberto. As amostras tratadas com permanganato de potássio, e substituindo o agregado miúdo em fração volumétrica de 10% apresentaram os resultados mais próximos as amostras de controles para todos os ensaios realizados. O GTR tratado causou aumento de 21% na resistência à compressão e 27% na resistência à flexão. A destinação dos pneus inservíveis para utilização como agregado reciclado em compósitos cimentícios reduz o descarte inadequado do produto, contribuindo para o desenvolvimento sustentável em seus 3 pilares, ambiental, social e econômico, e ainda contribui para as Objetivos de Desenvolvimento Sustentável (ODS) 3, 6, 11, 12, 13 e 15 da Agenda 2030.

Palavras-chave: argamassa emborrachada; agregado reciclado; pneu inservível; tratamento químico, agenda 2030; sustentabilidade.

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LIST OF ABBREVIATIONS

GTR – Ground tire rubber

IRSG – International Rubber Study Group

VA – Vinyl acetate

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1. INTRODUCTION

1.1. Problem Statement

The natural degradation process of vulcanized rubber, including tires, is slow due to the presence of crosslinks between rubber chains, stabilizers, and other additives. Additionally, a significant amount of valuable rubber resources is being wasted without proper recycling (PACHECO et al., 2014; PAULO; SARON, 2019; RECICLANIP, 2021; TORRETTA et al., 2015).

There are several methods available for recycling waste tires. One approach involves crushing the tires to produce rubber crumbs and powder, which can then be incorporated into polymeric or cementitious composites as recycled aggregate. Another technique, known as rubberized asphalt or Ecological Asphalt, utilizes rubber powder derived from crushed pneumatic waste as a filler in road paving mixtures. Additionally, researchers have explored the potential of utilizing this type of waste in the production of concrete bricks for the construction industry (BAUER et al., 2015; CAMPBELL-JOHNSTON et al., 2020; LANDI et al., 2018; NOHARA et al., 2005; SIDDIKA et al., 2019).

The mechanical strength of rubberized composites can be enhanced by enhancing the adhesion between cement paste and rubber particles. Several studies have explored different methods to achieve this, including pre-treatment of rubber through immersion in an alkaline solution of NaOH or sulfur-based petroleum residue (CS_2), water soaking, exposure to UV radiation, among others. These approaches aim to improve the bonding between rubber and cement, thereby enhancing the overall strength of the composite materials. (ABOELKHEIR et al., 2021b; LI et al., 2019; ROYCHAND et al., 2020; SIDDIKA et al., 2019; YOUSSEF et al., 2019).

Rubber, when used as a recycled aggregate, has the potential to impart additional properties to cement-based mixtures, including enhanced ductility, damping ratio, and energy dissipation. However, further investigation is required to better understand other properties that arise from the inclusion of this type of aggregate, such as the rheological and dynamic properties of hydration following its incorporation. These aspects warrant thorough study to fully comprehend the effects and optimize

the performance of rubberized cement-based mixtures (GERGES; ISSA; FAWAZ, 2018; SU et al., 2015; SUKONTASUKKUL; JAMNAM, 2013).

Following this reasoning, the present project aims to prepare floor boards with cementitious mortar containing tire rubber residue treated by an oxidizing agent, KMnO_4 or H_2O_2 , incorporating it into the cementitious matrix by volumetric replacement of fine aggregate in up to 15% (v/v). This project starts by washing the rubber with a NaOH solution to remove possible impurities adhered to its surface, as recommended in the literature. Then, the process of treatment by oxidizing agents will begin.

The surface and molecular analyzes of treated and control samples will be studied by Fourier Transform Infrared Spectroscopy - Total Attenuated Reflection (FTIR-ATR), in addition to contact angle and degree of crosslinking. The best treatment agent will be used in the continuation of the study to treat the rubber surface to be inserted into the cement matrix. The dosing process and adjusting the mortar proportions must consider the presence of treated and untreated rubber to adjust the amount of superplasticizer additive and the hydration water.

Finally, the mortar specimens will be molded and cured and stored until the date of each test; compressive strength and flexural tensile strength, total water absorption and void ratio, as recommended by the standards current.

The thermal properties of the floor boards are intended to be studied by monitoring the surface temperature changes on their surface while exposed to sun. The monitoring of the surface temperature of the floor boards is necessary to prevent potential accidents for users in areas exposed to solar radiation, for example, in applications such as poolside areas where people might be barefoot. Additionally, given the insulating nature of rubber, it is possible to achieve reductions in heat transfer from solar radiation in roofing systems for indoor spaces, thus reducing energy consumption in environments with mechanical air conditioning.

1.2. General Objective

To prepare floor boards with cementitious mortar containing tire rubber residue treated with an oxidizing agent, KMnO_4 or H_2O_2 , incorporating it into the cementitious matrix by the volumetric replacement of fine aggregate.

1.3. Specific Objectives

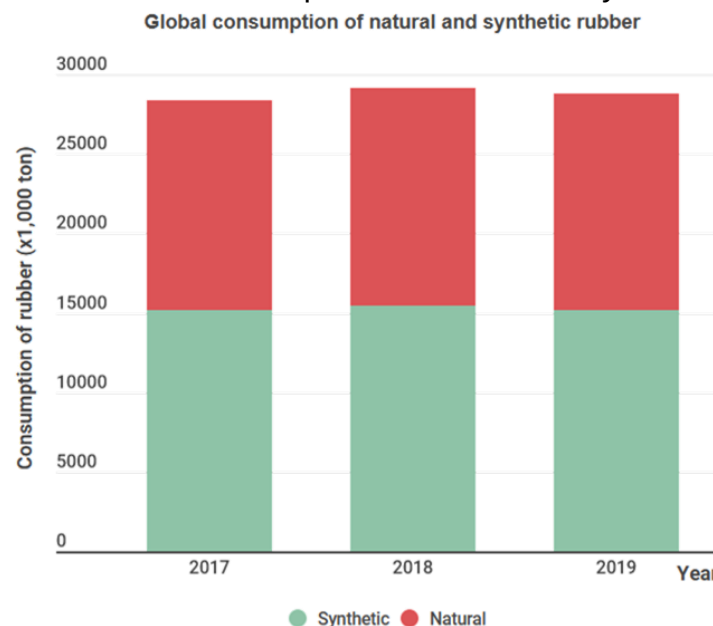
- a.** To wash the crushed rubber in an alkaline NaOH solution and then separately apply two oxidizing agents, KMnO_4 and H_2O_2 solutions, separately, as the chemical treatment of the crushed rubber;
- b.** To characterize the surface treated and untreated rubber, to evaluate the oxidation effect of each oxidizing agent, through the techniques of crosslinking degree, FTIR-ATR, and contact angle;
- c.** To define the best rubber treatment and adjust the dosage of mortar proportions with treated and untreated rubber;
- d.** To characterize the mortar specimens in terms of compressive strength, flexural tensile strength, total water absorption and void ratio;
- e.** To explore the thermal behavior of the rubberized mortars in the floor boards by monitoring the surface temperature changes on their surface while exposed to sun.

2. LITERATURE REVIEW

2.1. Global rubber scope

The International Rubber Study Group (IRSG) is an inter-governmental organization that plays a vital role in addressing global rubber-related issues. It promotes transparency in the world rubber market by providing up-to-date data on global rubber consumption (IRSG, 2020). Figure 1 present the global consumption of natural and synthetic rubber from 2017 to 2019, illustrating the distribution of over 28 million tons of rubber in the international market annually. However, the significant consumption of rubber-based products also results in a high discard rate, leading to various impacts on human health and the environment.

Figure 1 – Global consumption of natural and synthetic rubber



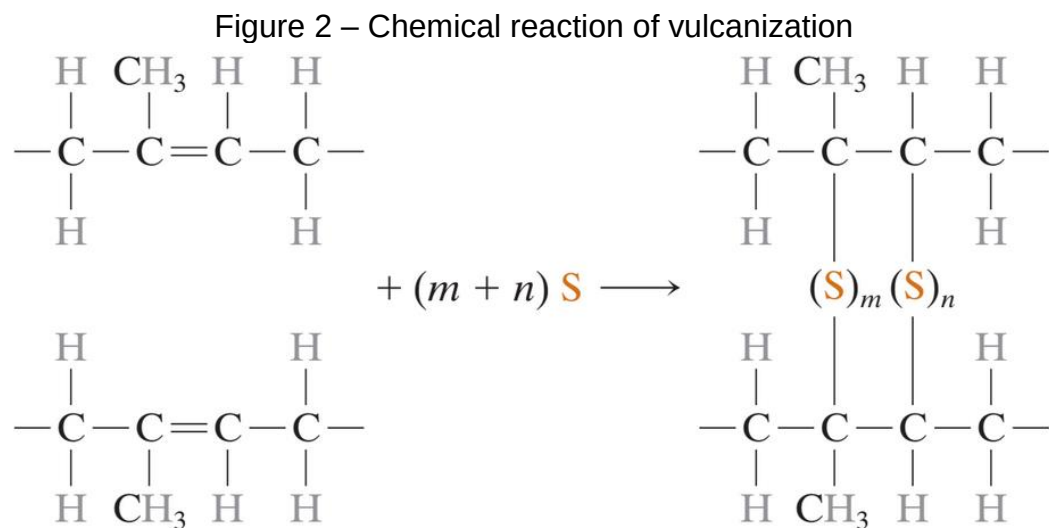
Source: adapted (IRSG, 2020)

The automotive and aerospace industries play a significant role in global rubber consumption as rubber serves as a crucial raw material for tire manufacturing (ABOELKHEIR, 2019). However, the disposal of end-of-life tires poses a considerable challenge, as evidenced by the staggering figure of 1 billion discarded tires per year, with 50% of them remaining untreated (THOMAS; GUPTA; PANICKER, 2016). In Brazil, Reciclanip, an initiative by the tire industry, reported that nearly 42 million end-

of-life tires were disposed of in 2020, and as of April 2021, over 11 million had already been discarded (RECICLANIP, 2021).

2.2. Rubber and vulcanization

Vulcanization is a cheap and fast process to improve the performance of rubber throughout the addition of sulfur to the elastomer in high temperature via a non-reversible chemical reaction (Figure 2), where “m” and “n” sulfur atoms crosslink the polymer chain (CALLISTER JR.; RETHWISCH, 2010; CANEVAROLO JR., 2006).



Source: (CALLISTER JR.; RETHWISCH, 2010)

The non-vulcanized rubber is a soft elastomer (CALLISTER JR.; RETHWISCH, 2010; RODGERS, 2004), therefore it is considered useless to conventional applications, such as in tires, shoes, etc. Crosslinking the polymeric chain improves the mechanical strength and introduces elasticity to the rubber turning it into a thermoset polymer, expanding its use (CANEVAROLO JR., 2006).

The process ensures the conversion of viscous and entangled molecules with long chains into a three-dimensional elastic network, which is chemically joined at various points along the chain. As shown in Figure 2, the polymer chains are represented by the lines and the crosslinks by the points (RODGERS, 2004).

Transforming the elastomer in a thermoset polymer turns it unable to be remolded by the simple heating. Although many benefits are attributed in terms of

application expansion, it also difficult the mechanical recycle, promoting environmental damages due to incorrect material disposal (PAULO; SARON, 2019).

2.3. Environmental impacts due to the incorrect tire discards

The global growing of the automobilist industry supported the production and sales of new tires, which resulted in huge piles of waste tires improperly discarded, promoting a potential environmental risk throughout the creation of landfills, sometimes irregulars (GANJIAN; KHORAMI; MAGHSOUDI, 2008; THOMAS; GUPTA; PANICKER, 2016). Tires residues are not biodegradable under normal conditions, because they are weatherproof. As a result, the improper tire disposal became a huge environmental concern for specialists, Figure 3 shows the biggest tire cemetery in the world, located in Sulaibiya, Kuwait, which is possible to be seen from satellites images (PACHECO-TORRES et al., 2018).

Figure 3 – Tires cemetery in Sulaibiya, Kuwait



Source: (GOOGLE, 2021)

Tires are vital items for the operation of vehicles, and with the increasing number of automobiles in the world, the demand for new tires, proportionally, require a larger consumption of raw material and further promote the generation of waste. Use of recycled rubber in the manufacture of new tires is impractical, due to its complexity,

arisen by the vulcanization process (PACHECO-TORRES et al., 2018; RECICLANIP, 2021).

The most part of discarded end-of-life tires are stocked without any specific treatment. The growing amount of landfills presents many phytosanitary risks, increasing the chances of fires, mainly during summer (CARMO; SILVA, 2009). Also, millions of tires are buried or burned around the world (ZHANG et al., 2009). It is estimated that for each burned tire, almost 10 liters of oil are thrown on the ground, generating groundwater pollution (CIMINO; ZANTA, 2005).

Rubber wastes are characterized as toxic and dangerous products. The disposal of tires by different forms, such landfill, burning, fuel use, pyrolysis, and many others, has strong environmental, economic and social impact, through the air, water and soil pollution, reverberating directly on public health, and soil of developed and non-developed countries, (PACHECO-TORRES et al., 2018; THOMAS; GUPTA; PANICKER, 2016).

Discarded tires may, also, store water and organic wastes for a long period due to their particular shape and impermeable nature, providing a favorable habitat for breeding mosquitoes, and various pests, especially *Aedes Aegypti*, which is responsible for diseases such as dengue, yellow fever and chikungunya (CARMO; SILVA, 2009; THOMAS; GUPTA; PANICKER, 2016).

2.4. Laws and regulations

According to Associação Nacional da Indústria de Pneumáticos (ANIP) (2021) data, around of 67,9 million of tires unity were sold in Brazil in 2020. Reciclanip is the biggest responsible for appropriate tires disposal in Brazil, and it was created and managed by the companies Bridgestone, Michellin, Pirelli and Goodyear (RECICLANIP, 2021).

In Brazil, due to the illegal growing of tire disposal, CONAMA (Conselho Nacional do Meio Ambiente) decreed the resolution nº 416/2009, abolishing the resolutions nº 258/1999 and 301/2002, trying to solve the problem around the country, promoting the appropriated and proper disposal of tires.

CONAMA 416/2009 resolution establishes the reverse logistic concept to tires, and attributed to the manufacturers the responsibility to discard end-of-life tires, whilst

the costumer, also, is responsible to send the material to the indicated units by the manufacturers when it is useless (CONSELHO NACIONAL DO MEIO AMBIENTE, 2009).

Environmental and Natural Renewable Resources Brazilian Institute (IBAMA) indicates that Brazil did not complete the end-of-life tires correct disposal goals in 2019, it was expecting around of 600.000 ton of tires, and only 585.000 ton were correctly discarded, remaining 15.000 ton disposed inappropriately, which may cause damages to the environment and the health. The correct disposed wastes were reused following the information under Figure 4, and the co-processing was the main method:

Figure 4 – Brazilian tires disposal technologies



Source: (ALVES, 2021)

The most used techniques to reuse the rubber were co-processing and granulation. Co-processing is a technique which uses high temperatures to degrade the tires and generates heat to cement kilns. The method is a cheaper alternative to the cement manufactures when compared to conventional fuels (GOBBI, 2002). Granulation consists in separating rubber, steel and nylon from the tire and crush them, separately. The generated material is used as raw material in cement industries, shoes manufactures, etc (BAUER et al., 2015; NOHARA et al., 2005).

2.5. Rubber waste management

The irregular urban population growing over the years is the main responsible of global environmental issues, due to the accelerated and unplanned industrialization, which promoted the irresponsible use of natural resources, and generation of waste growing exponentially and proportionally to the consumption (MACÊDO; MARTINS, 2015).

Many countries have their own solid waste management policies, and the Brazilian National Solid Waste Policy defines solid waste as material, substance, object or good discarded arising from human activities in society, found in solid or semi-solid state, as well as gases contained in containers and liquids that require prior treatment before being dumped into water courses (BRASIL, 2010).

Also, Brazil follows the NBR 10.004 standard, which classifies solid wastes as described in Table 1 – Solid wastes according to NBR 10.004 standards classification.

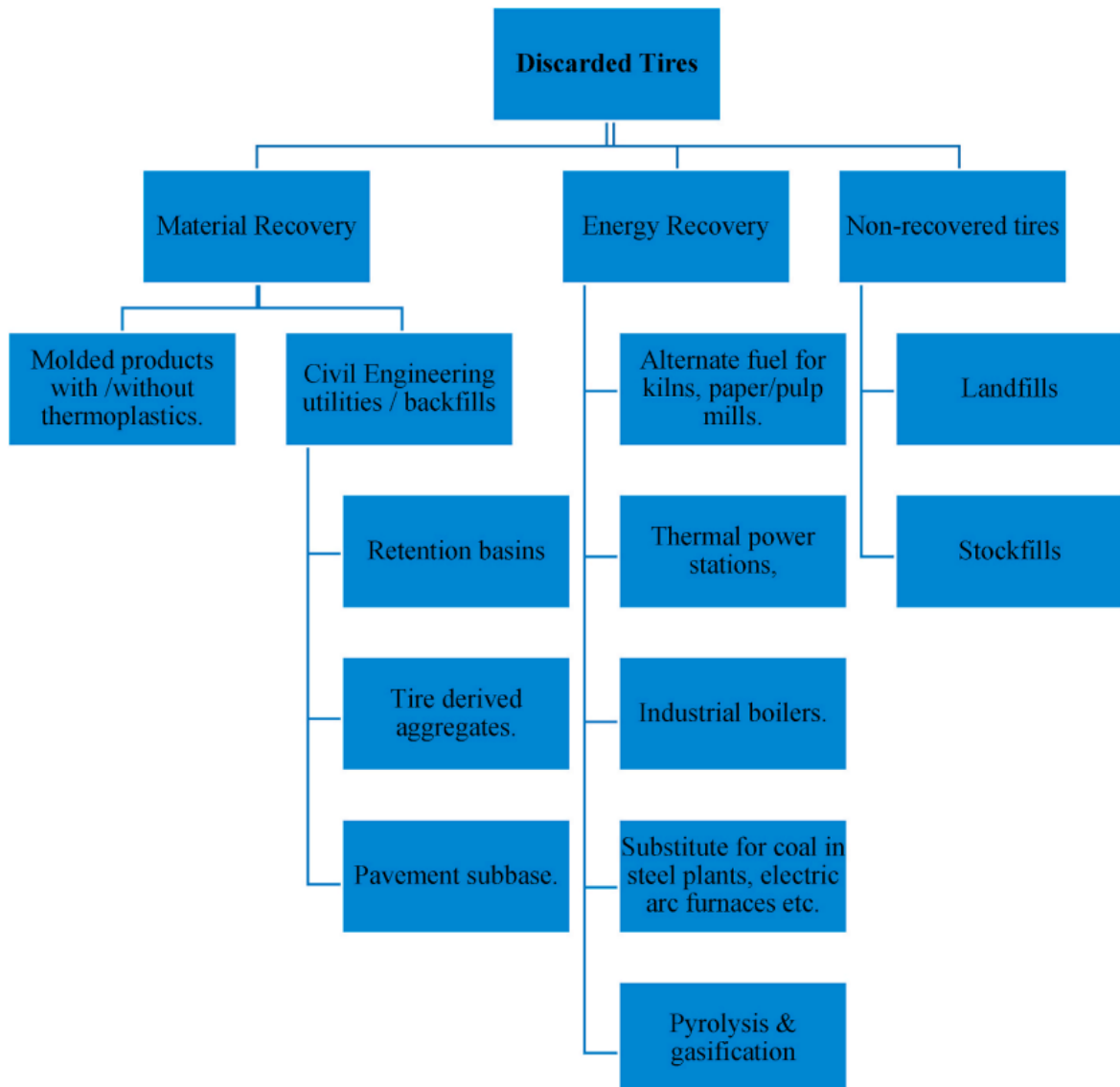
Table 1 – Solid wastes according to NBR 10.004 standards classification	
TYPE	DESCRIPTION
Class I – Hazardous wastes	Present a risk of danger, having characteristics of corrosion, flammability, pathogenicity, reactivity or toxicity.
Class II – Non-hazardous wastes	Not qualified as hazardous (Class I) and are divided into: class IIA – non inert and class IIB – inert.
Class IIA – Non-inert wastes	Not hazardous, but are not inert, may have properties such as biodegradability or water solubility.
Class IIB – Inert wastes	When submitted to solubility tests, none of their solubilized constituents showed concentrations higher than the standards of potability of water.

Source: (ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS, 2004, p. 100)

According to the NBR 10.004, tires are classified as Class IIA (non-inert waste), and the standard use to define it as undesirable wastes due to the high metals content, as zinc and manganese, in the solubilized extract.

There are many options to reuse end-of-life tires, and energy recovery, material recovery and civil engineering recovery are the most common destiny to them (see Figure 5).

Figure 5 – Different recycling strategies currently adopted for tire recovery.



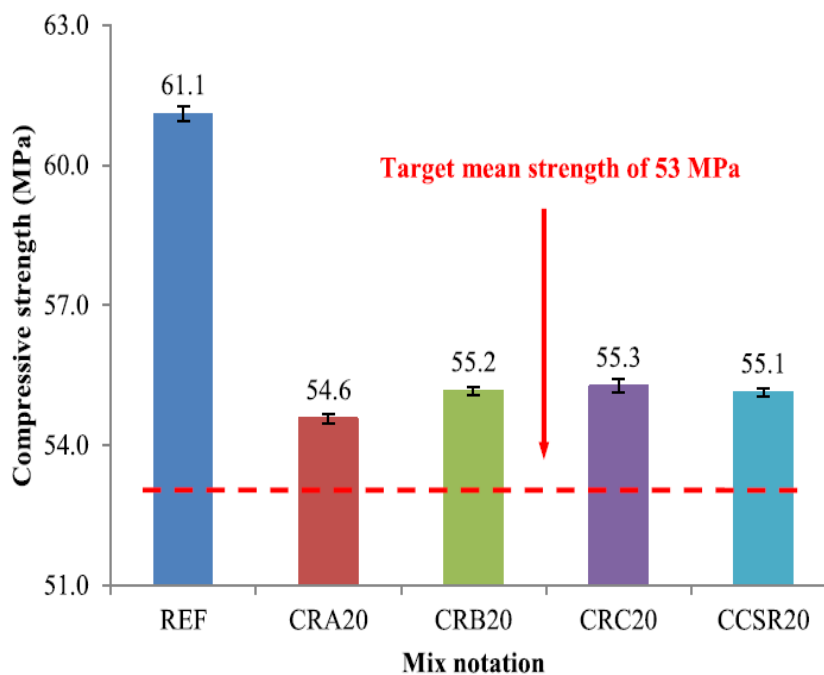
Source: (HAMDI; ABDELAZIZ; FARHAN, 2021)

2.6. Mechanical properties of rubberized cementitious composites

2.6.1. Compressive strength

Compressive strength is one of the most important properties of cementitious composites, and it depends on the physical and mechanical characteristics of their constituents. However, some researchers indorse that replace these materials by rubber reduces the compressive strength of cementitious composites (ABOELKHEIR, 2019; GESOĞLU et al., 2014; MEDINA; MEDINA; HERNÁNDES-OLIVARES, 2014; ROYCHAND et al., 2020; THOMAS; GUPTA; PANICKER, 2016), as seen in Figure 6, where REF is the reference sample, and the others are different mixtures proportions of concrete. REF – Reference Concrete; CRA – Concrete Rubber A (rubber grounded to 3mm); CRB – Concrete Rubber B (rubber grounded to 0.5mm); CRC – Concrete Rubber C (rubber grounded to 0.3mm); CCSR – Blending of RA, RB and RC; Number 20 represents de amount of rubber used to replace the fine aggregate by volume (20%) (SU et al., 2015). The reasons for compressive strength reduction on rubberized cementitious composites are the weak adhesion between rubber particles and cement paste, and the stiffness difference of both (ABOELKHEIR, 2019; SHU; HUANG, 2014; SI; GUO; DAI, 2017).

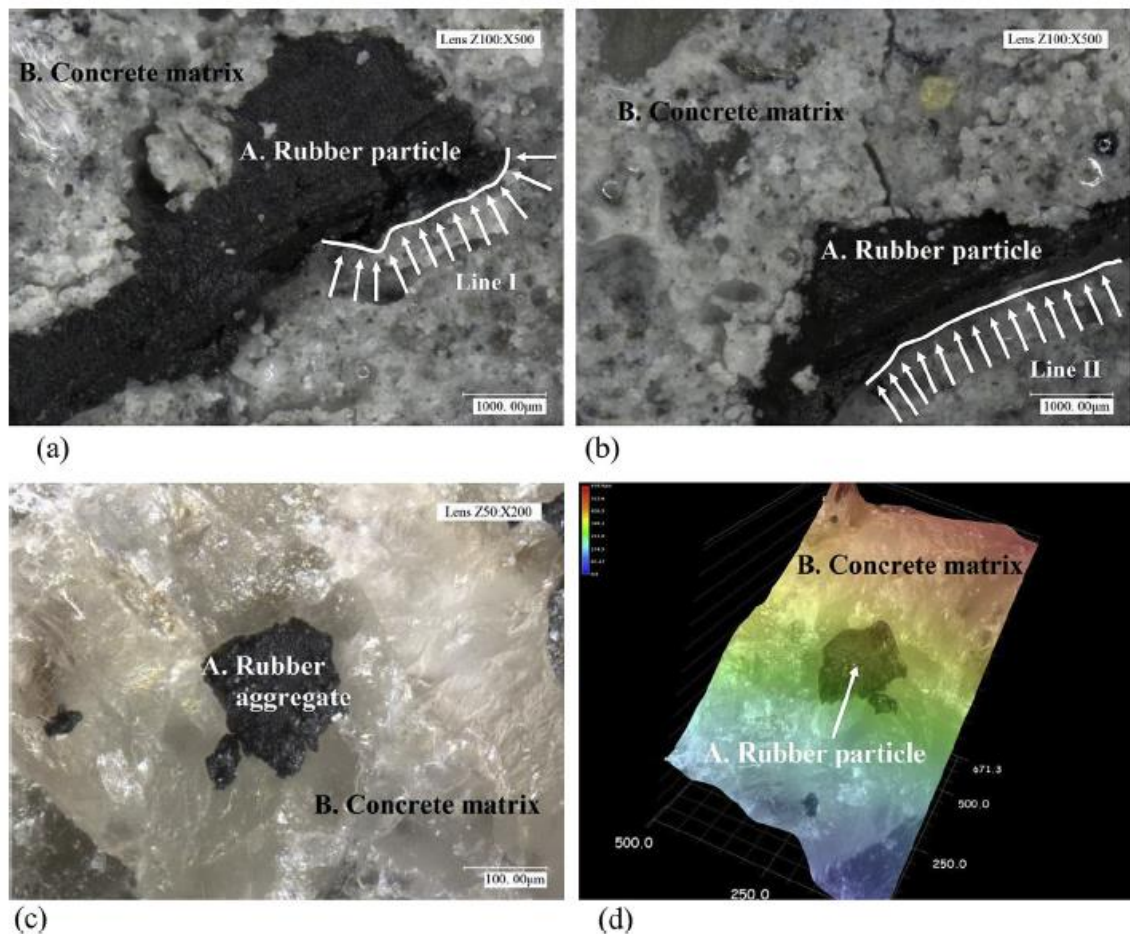
Figure 6 – 28 days compressive strength of rubberized cement-based composites.



Source: (SU et al., 2015)

Weak adhesion between rubber particles and cement paste is justified by the hydrophobic nature of rubber, which is responsible for a poor cement hydration around the rubber aggregate, and it generates a non-uniform distribution of stress on composite (HUANG; SHU; CAO, 2012). Su et al. (2015) studied the compressive strength of rubberized concretes, and according to the previous affirmation, noted that the rubber-matrix interfacial behavior is responsible for the negatives results about compressive strength (see Figure 7).

Figure 7 – Micrographs of rubber-matrix interface



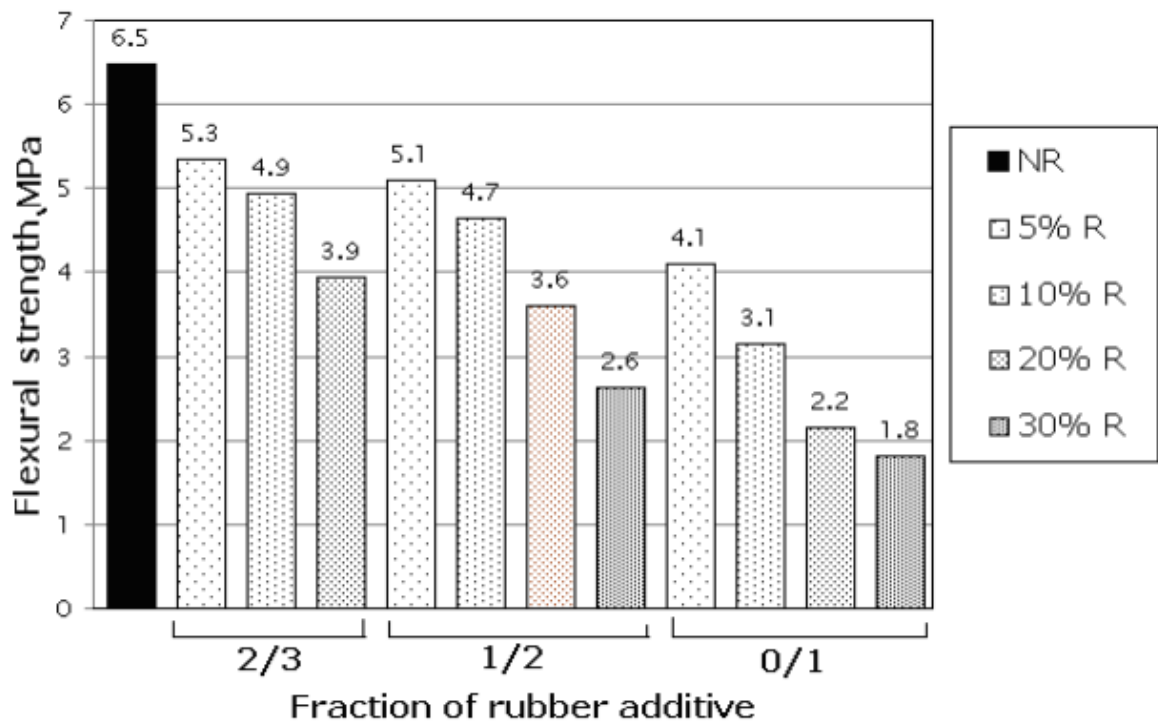
Source: (SU et al., 2015)

Also, the stiffness of rubber aggregates is lower than cement paste, thus, cracks develop fast around de rubber particles during loading, and they grow on all matrix, causing an accelerated fracture of cement composite (GANJIAN; KHORAMI; MAGHSOUDI, 2008).

2.6.2. Flexural strength

The flexural strength of rubberized cementitious composites samples decreases as the aggregate replacement volumetric fraction increases, as shown in Figure 8 (KHATIB; BAYOMY, 1999; MEDINA et al., 2017; NAJIM; HALL, 2013; ROYCHAND et al., 2020; SKRIPKIUNAS; GRINYS; MIŠKINIS, 2009).

Figure 8 – Comparison of flexural strength of concrete with different mass fractions of fine aggregate replacement by rubber



Source: (SKRIPKIUNAS; GRINYS; MIŠKINIS, 2009)

Khatib et al. (1999) realized that flexural strength decreasing is proportional to the compressive strength test, and rubberized concrete samples had higher deflection before failure than reference mixture. The higher deflection is due to the rubber stiffness, which overcome the natural aggregate. The rubber particle size promotes changes to the flexural strength of samples, as observed by Skripkiunas et al. (2009), where the finest particles generate less decreases than coarse particles.

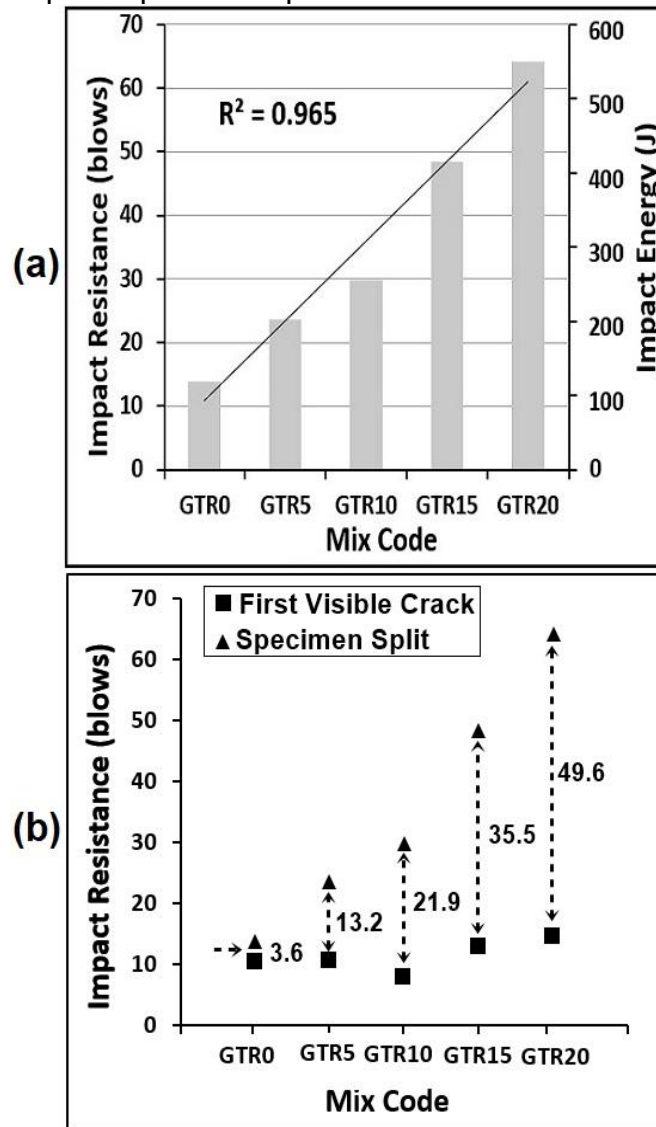
2.6.3. Impact resistance

Rubber aggregate improves the ability of concrete to absorb and dissipate energy (GERGES; ISSA; FAWAZ, 2018; MEDINA et al., 2017; SUKONTASUKKUL; JAMNAM, 2013). The higher ductility and toughness of rubber particles are responsible to ensure a deformable and soft surface on the composite (ABOELKHEIR, 2019; MEDINA et al., 2017), acting as springs without failure (YOUSSF; HASSANLI; MILLS, 2017).

Thus, impact resistance is one of the most important property of rubberized cementitious composites, allowing many applications where is requested increased fracture energy, such as bulletproof panels, pavements, and others (ABOELKHEIR, 2019; SUKONTASUKKUL; JAMNAM, 2013).

Furthermore, the fracture energy of rubberized cementitious composites is directly related to the applied rubber particle size (ROYCHAND et al., 2020). Aboelkheir (2019) performed the drop weight impact test, according to ACI 544.2R-89 modified, in rubberized concrete paving samples, which were made with GTR (see Figure 9) and reported an increase in the absorbed impact energy proportionally to the amount of rubber replacement.

Figure 9 – (a) Influence of GTR on the impact resistance and energy of conventional and rubberized concrete paving units; (b) Required blows to cause a complete specimen split after the first visible crack

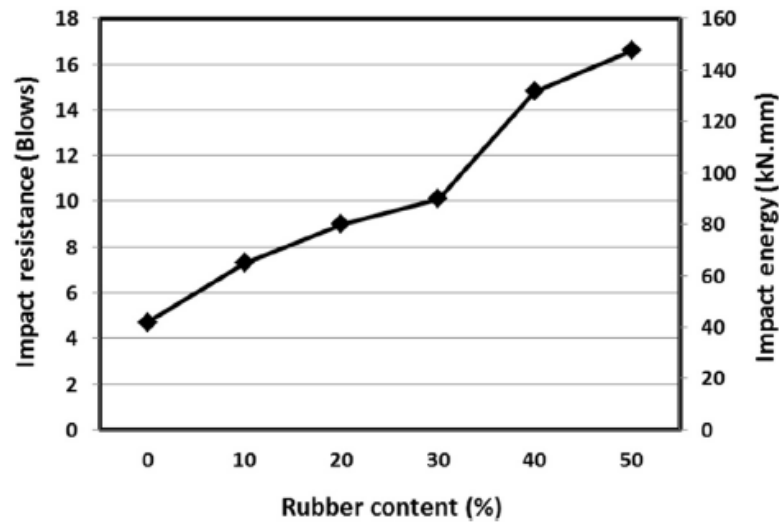


Source: (ABOELKHEIR et al., 2021b)

Many authors reported that rubber aggregate does not delay the cracking propagation on cement composites, and its behavior is similar to the conventional concrete (ABOELKHEIR, 2019; GERGES; ISSA; FAWAZ, 2018), as shown in Figure 9-b.

Youssf et al. (2017), also, performed a drop weight test with rubberized concrete, where they replaced the sand by rubber particles, and reported similar results to the other authors (see Figure 10).

Figure 10 – Effect of rubber content on concrete impact resistance

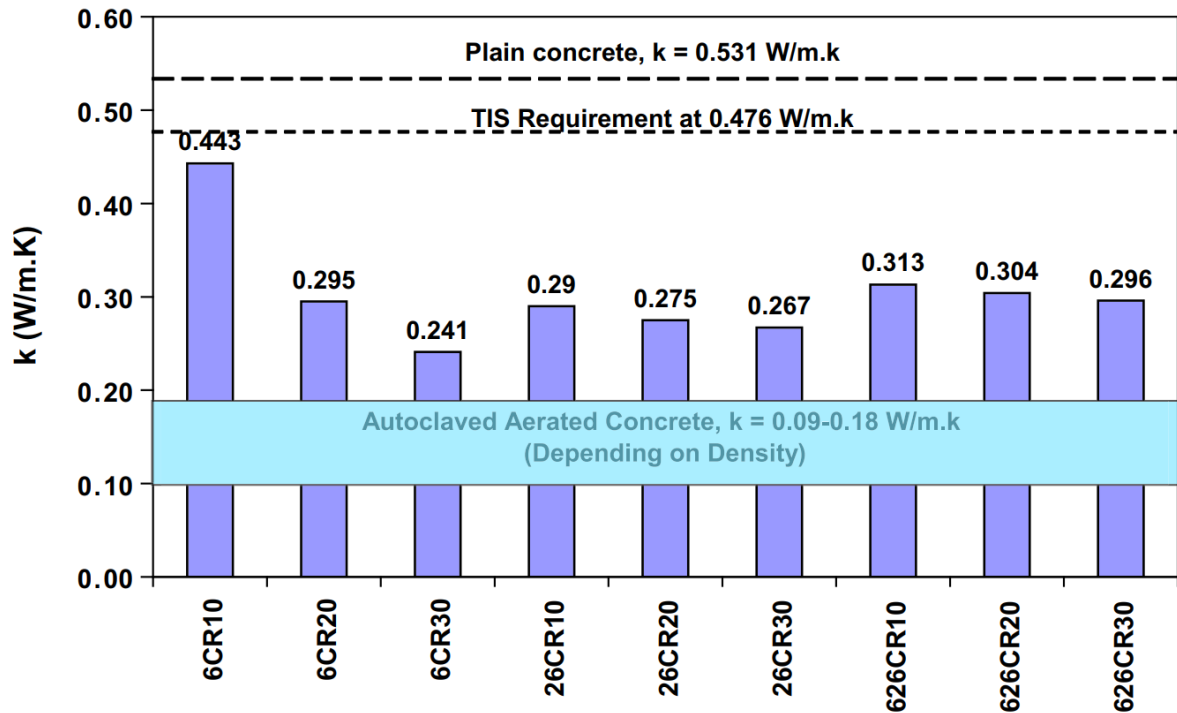


Source: (YOUSSEF; HASSANLI; MILLS, 2017)

2.7. Thermal properties of rubberized cementitious composites

The thermal conductivity of rubberized cementitious composites tends to decrease when the rubber replacement volume fraction increases (BENAZZOUK et al., 2008; MEDINA et al., 2017; SUKONTASUKKUL, 2009). In addition, it is possible to observe the influence of the particle size on the thermal properties of rubberized cementitious composites. Figure 11 shows the rubber used as partial replacements of the fine aggregate using volumetric fraction of 10%, 20% and 30%, and the code to identify the sample is MESH + CR (crumb rubber) + Volumetric Fraction. Sukontasukkul (2009) performed the analysis of thermal conductivity on samples with different granulometry of rubber, mesh #6 and mesh #26.

Figure 11 – Thermal conductivity of crumb rubber concrete compared with conventional concrete and autoclaved aerated concrete

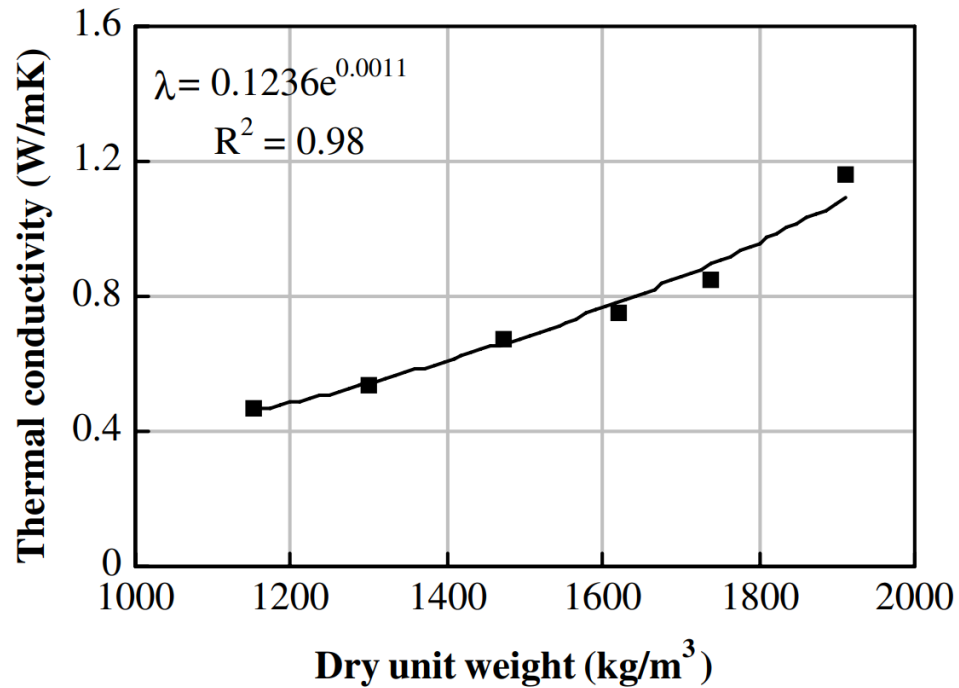


Source: (SUKONTASUKKUL, 2009)

The thermal conductivity coefficient (k) reduction proportionally to the amount of rubber in mixture occurs due to two reasons: rubber promote the increase of entrapped air in samples, and the rubber thermal conductivity is lower than the usually used natural coarse aggregate (MEDINA et al., 2017). Rubber aggregate changes the cementitious composite rheology, making the mixing and curing process more difficult, which may entrap air bubbles to the material, increasing the porosity and hence the k values are reduced.

It is possible to observe the exponential behavior between thermal conductivity and bulk density of rubberized cementitious composites (see Figure 12), which happens due to the amount of rubber in mixture, replacing the natural aggregate.

Figure 12 – Relationship between thermal conductivity and dry unit weight of rubberized cementitious composite

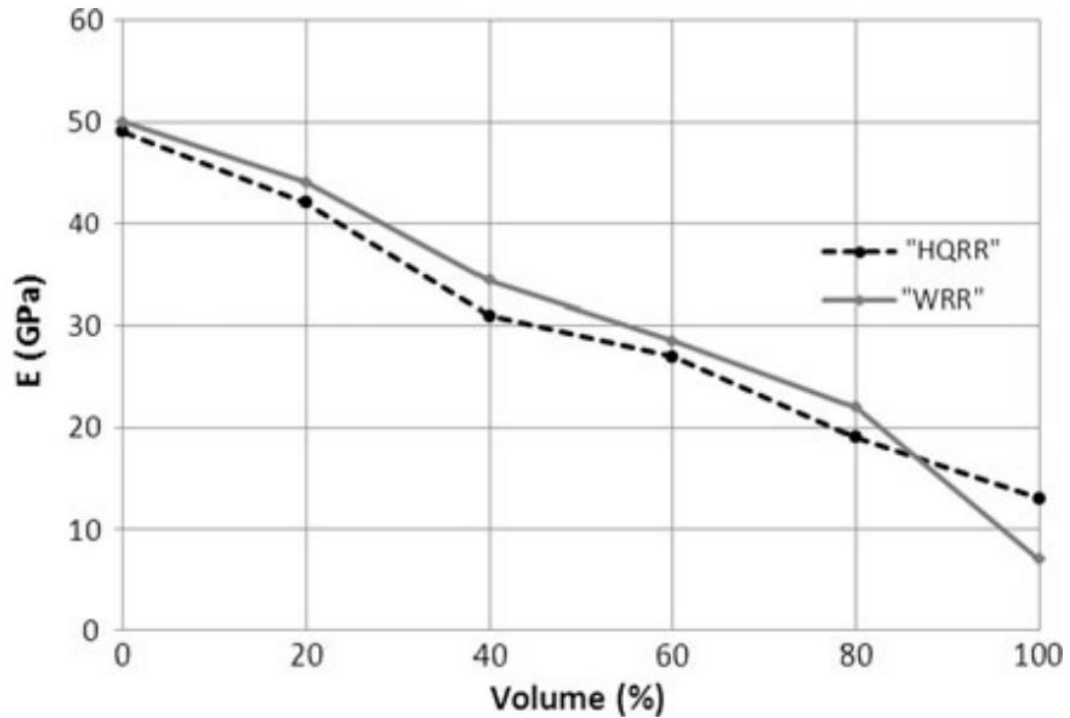


Source: (BENAZZOUK et al., 2008)

2.8. Dynamic Young modulus

Dynamic Young modulus of rubberized cementitious composites is reduced as the aggregate volume replacement increases by GTR (see Figure 13) (GUO et al., 2017; MEDINA; MEDINA; HERNÁNDES-OLIVARES, 2014). Low strength and elastic modulus of rubber are the responsible for the reduction in dynamic elastic modulus of the rubberized cement-based composites (ATAHAN; YÜCEL, 2012).

Figure 13 –Ultrasonic Young modulus and replaced ratios of rubber instead of stone aggregate.



Source: (MEDINA; MEDINA; HERNÁNDES-OLIVARES, 2014)

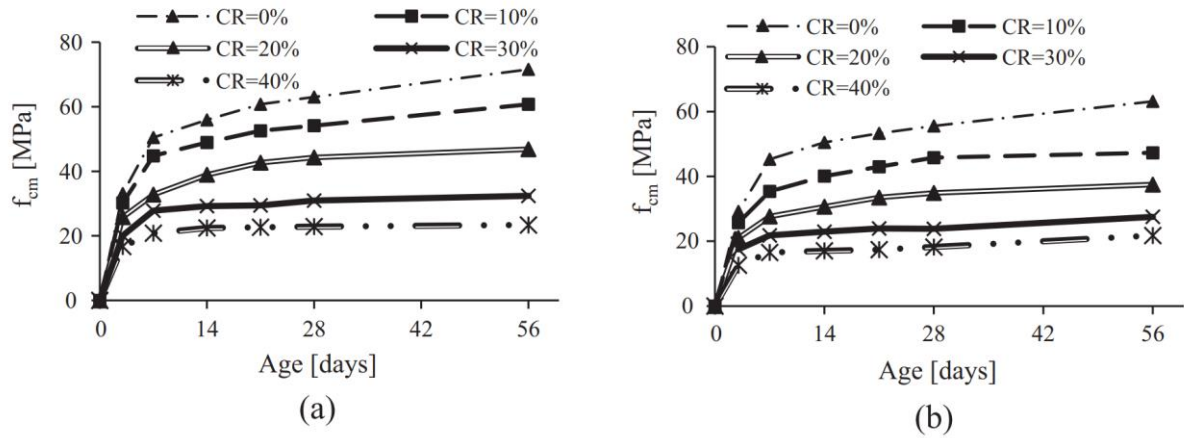
2.9. Rubber recovery and treatment techniques

2.9.1. Water soaking

The water soaking treatment to rubberized aggregates is one of the first techniques of elastomer recovery to apply in cementitious composites (ELDIN; SENOUCI, 1994). Although it is a simple technique, it does not presents significant enhancement to the rubber/cement interaction (MOHAMMADI; KHABBAZ; VESSALAS, 2014; NAJIM; HALL, 2013; RICHARDSON; COVENTRY; WARD, 2012; YOUSSEF et al., 2018).

Figure 14 presents compressive strength test results at different ages of the rubberized concrete, where the rubber aggregate was soaked in water solution, and it was used to replace the sand by different volumetric fractions.

Figure 14 – Effect of aging on compressive strength of crumb rubber (CR) concrete: (a) WC = 0,40 and (b) WC = 0,45.



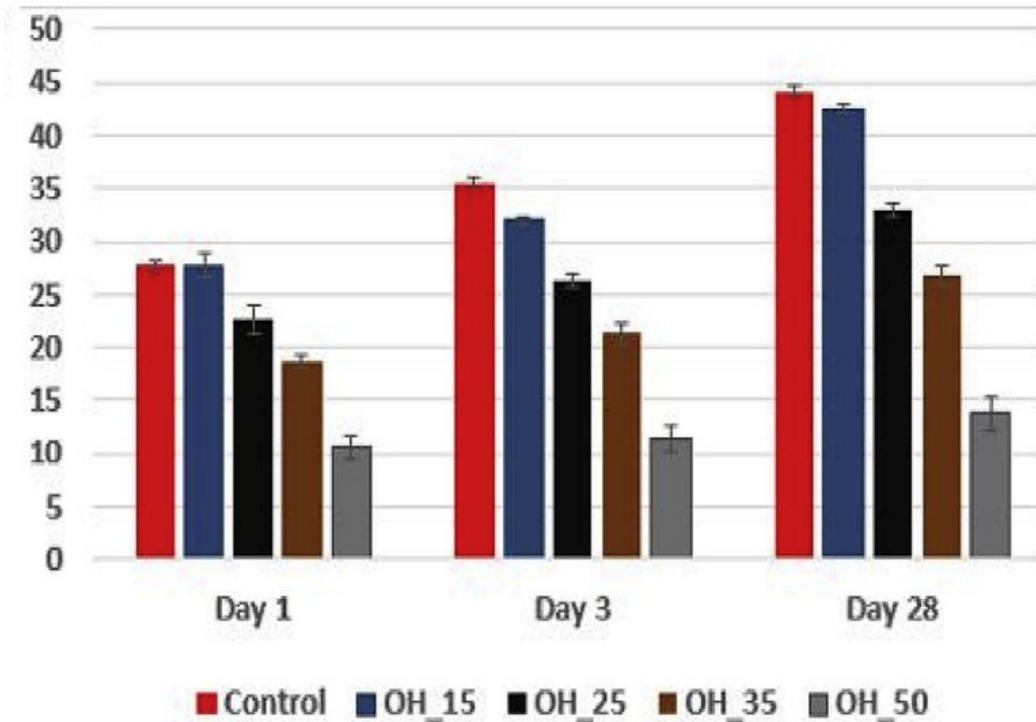
Source: (MOHAMMADI; KHABBAZ; VESSALAS, 2014)

2.9.2. NaOH

Rubber surface treatment using aqueous solution of sodium hydroxide (NaOH) promotes improvements to the durability (SI; GUO; DAI, 2017), fracture energy and flexure strength (SEGRE; JOEKES, 2000). Otherwise, the compressive strength impacts due to this treatment are small when the fine aggregate volume replacement average by rubber increases (MARQUES et al., 2008; YOUSSEF et al., 2019).

The literature demonstrates that end-of-life tires treated particles using sodium hydroxide, with diameter range of 0,075-4,75mm is viable to enhance the mechanical properties of rubberized cementitious composites that uses it to replace the fine aggregate. Figure 15 shows the compressive strength of rubberized composites where the rubber particles were treated by immersion in 1N NaOH for 40 minutes, and then it was washed with water until its pH becomes neutral (GUO et al., 2017; KASHANI et al., 2018; MOHAMMADI; KHABBAZ; VESSALAS, 2016; YOUSSEF; ELGAWADY; MILLS, 2016).

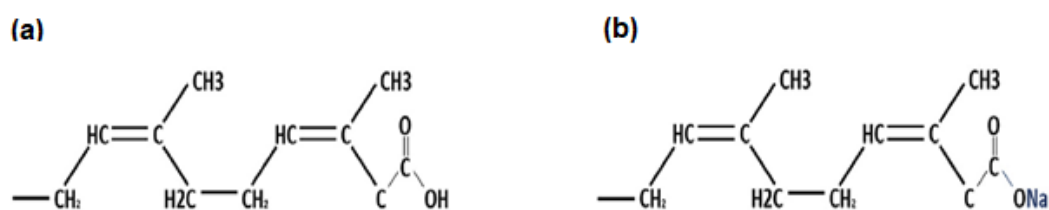
Figure 15 – Compressive strength of rubberized concrete samples using rubber aggregate treated using NaOH replacing the fine aggregate by volume average.



Source: (GUO et al., 2017)

Chemically, the presence of non-polar long polymeric chains containing non-polar groups enhances the lack of polarity on the rubber surface. The carboxylic acid ion replacement by Na^+ on the polymer surface after its immersion in NaOH, explains the rubber hydrophobic nature rupture, as seen in Figure 16, and it changes the main elastomer component structure, the cis-polyisoprene, which promotes enhancements during the cement hydration (GUO et al., 2017; SU et al., 2015).

Figure 16 – Mechanisms to the NaOH solution surface treatment methods. (a) Cis-polyisoprene structure with the carboxylic acid group; (b) The reaction product of NaOH solution with cis-polyisoprene with carboxylic acid group.



Source: (GUO et al., 2017)

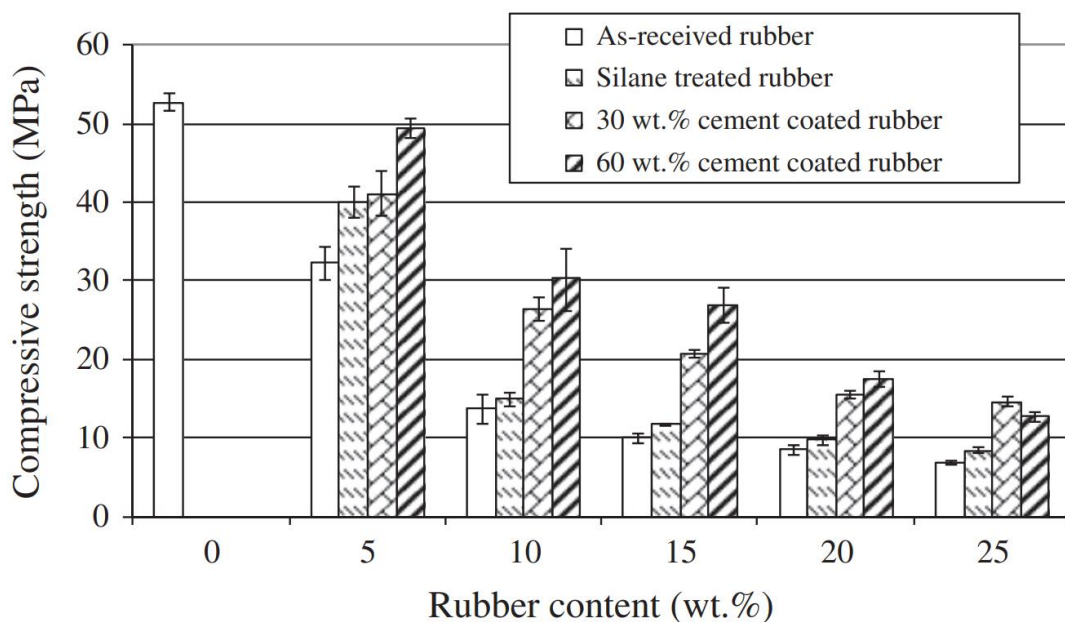
The NaOH immersion time is an important variable to the treatments performance and many authors studied the effect of time on rubber hydrophilicity highlighting 30 minutes as the optimal exposure time (KASHANI et al., 2017; YOUSSEF; MILLS; HASSANLI, 2016).

2.9.3. Silane couple agent

The silane couple agent rubber surface treatment ensures enhanced compressive strength of rubberized composites compared to the results of non-treated rubber aggregate (see Figure 17) (DONG; HUANG; SHU, 2013; HUANG; SHU; CAO, 2012; LIU; ZHANG, 2015), due to the chemical reaction combined with both organic and inorganic materials.

Likewise the other methods mentioned in this work, the silane couple agent surface treatment provides a better adhesion between rubber particle and cement paste, while it promotes the polymer hydrophobic nature rupture, which is responsible for the chemical weakness of interfacial transition zone (GUO et al., 2017; HUANG; SHU; CAO, 2012; LIU; ZHANG, 2015).

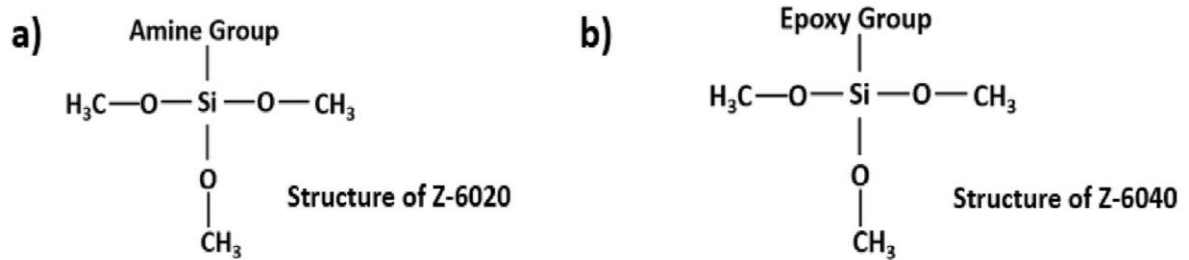
Figure 17 – Compressive strength of Portland cement concrete using rubber treated by silane couple agent



Source: (HUANG; SHU; CAO, 2012)

Different studies highlighted the use of the couple agents Z-6020 ($\text{H}_2\text{NCH}_2\text{CH}_2\text{NHCH}_2\text{CH}_2\text{Si}(\text{OCH}_3)_3$) and the Z-6040 ($\text{OCH}_2\text{CHCH}_2\text{OCH}_2\text{CH}_2\text{CH}_2\text{Si}(\text{OCH}_3)_3$) in mixture proportion of 1:1, where their structures are presented in Figure 18 (DONG; HUANG; SHU, 2013; GUO et al., 2017; HUANG; SHU; CAO, 2013).

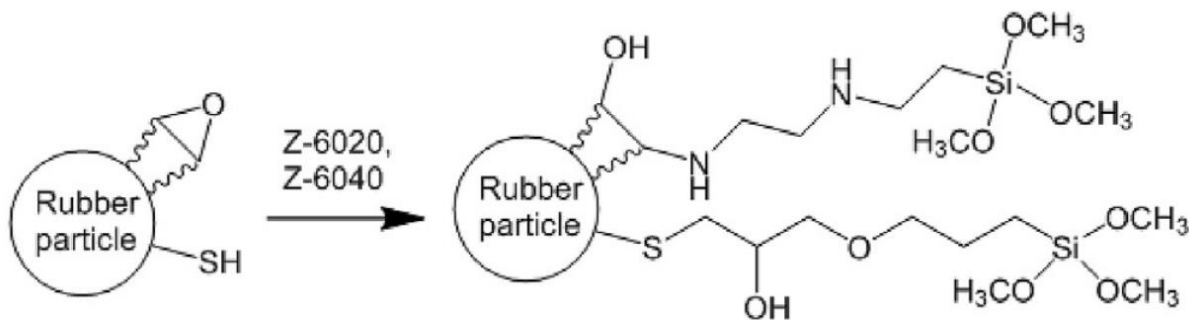
Figure 18 – Silane couple agent structures



Source: (GUO et al., 2017)

The amine in Z-6020 promotes the reaction between epoxy group of Z-6040 and epoxy contents on rubber particle surface (GUO et al., 2017), which allows the Z-6020 molecule attach to the epoxy terminal of Z-6040 molecule (DONG; HUANG; SHU, 2013; HUANG; SHU; CAO, 2013), as seen in Figure 19.

Figure 19 – Chemical reaction product structure of silane couple agent rubber surface treatment



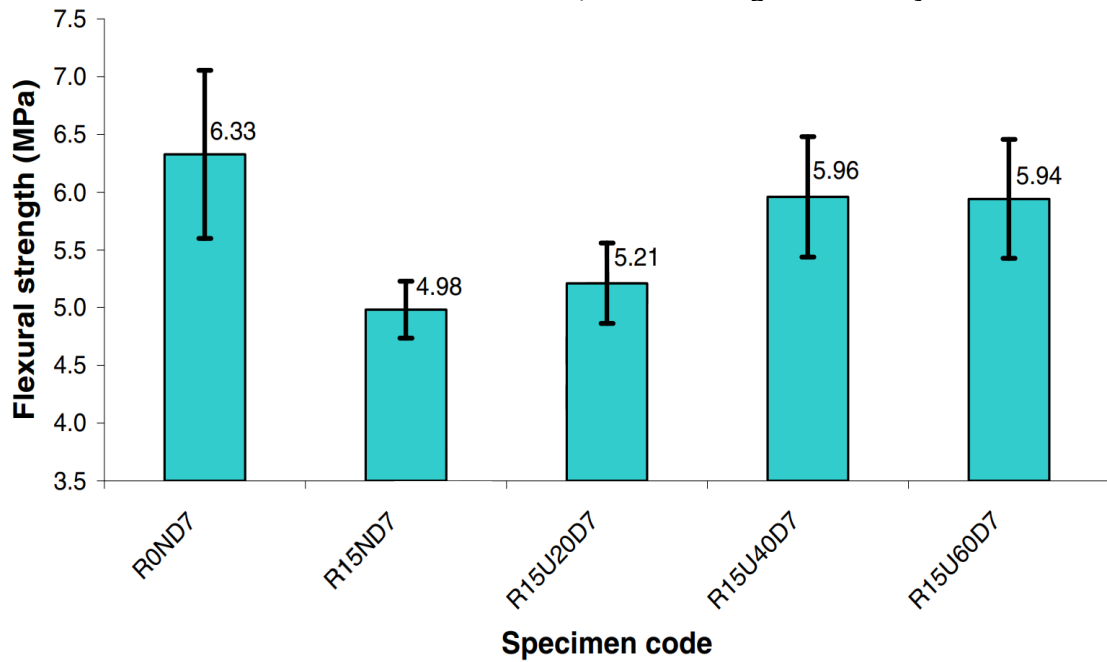
Source: (GUO et al., 2017)

2.9.4. UV radiation exposure

The UV radiation exposure rubber surface treatment promotes significant improvements to the mechanical properties of rubberized cementitious composites, as example, flexure strength, as shown in Figure 20, where Ossola and Wojcik (2014),

realized that until 15% of volumetric fraction of rubbers aggregate, the concrete samples presented enhanced mechanical properties, proportional to the UV radiation exposure time on rubber particles.

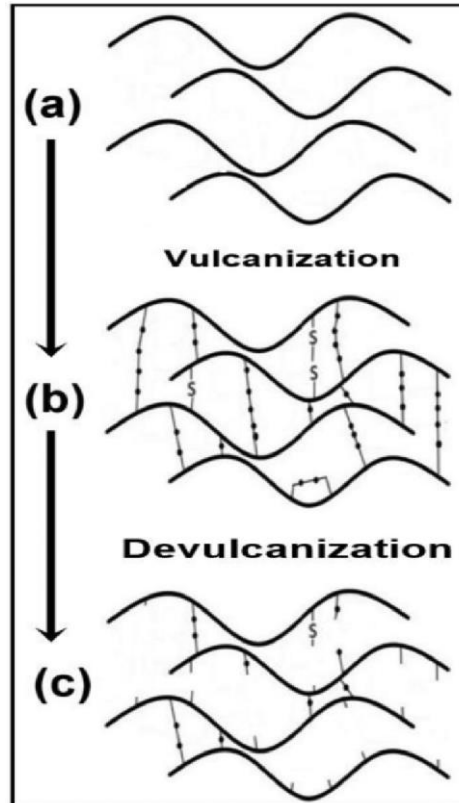
Figure 20 – Average flexural strength for each condition. R – Rubber; 0 or 15 – Amount of rubber used in volume fraction; N – No treatment; U20, U40 or U60 – UV treatment time in minutes; D7 – Curing time in days.



Source: (OSSOLA; WOJCIK, 2014)

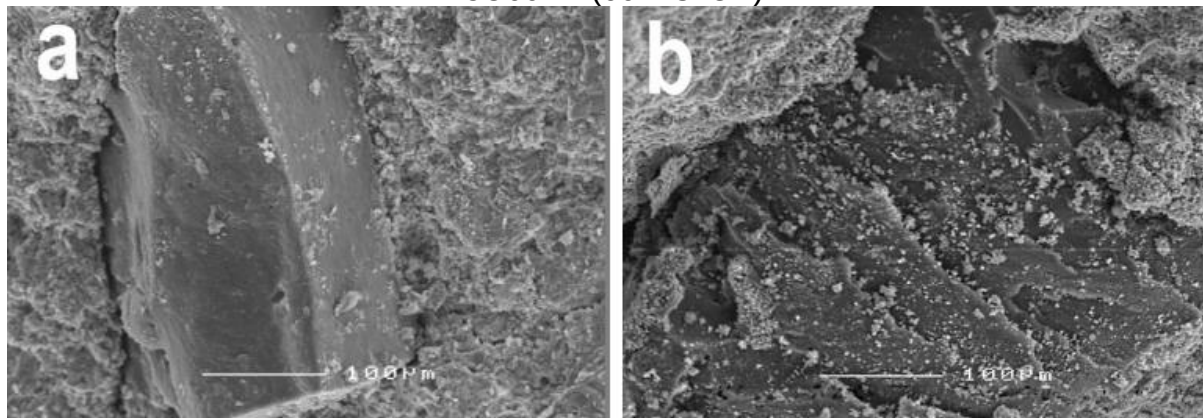
The UV radiation exposure tends to devulcanize the rubber particle, in order to reduce the crosslinked networks of the polymeric chain, as seen in Figure 21. The physicochemical effects of this treatment ensure improved hydrophilicity to the elastomer, whilst the water contact angle is reduced, and there is enhanced cement hydration on ITZ, as well. It can be observed in Figure 22-b that the cement particles could be precipitated on the rubber aggregate surface (OSSOLA; WOJCIK, 2014; SHANMUGHARAJ; KIM; RYU, 2005, 2006).

Figure 21 – Schematization of the vulcanization/devulcanization process: (a) non-vulcanized rubber; (b) vulcanized rubber and (c) devulcanized rubber.



Source: (ABOELKHEIR et al., 2021b)

Figure 22 – SEM images of specimens, all showing particulate matter (lighter spots) on embedded rubber particles (central objects): (a) R15ND7 (no UV) and (b) R15U60D7 (60 hrs. UV).



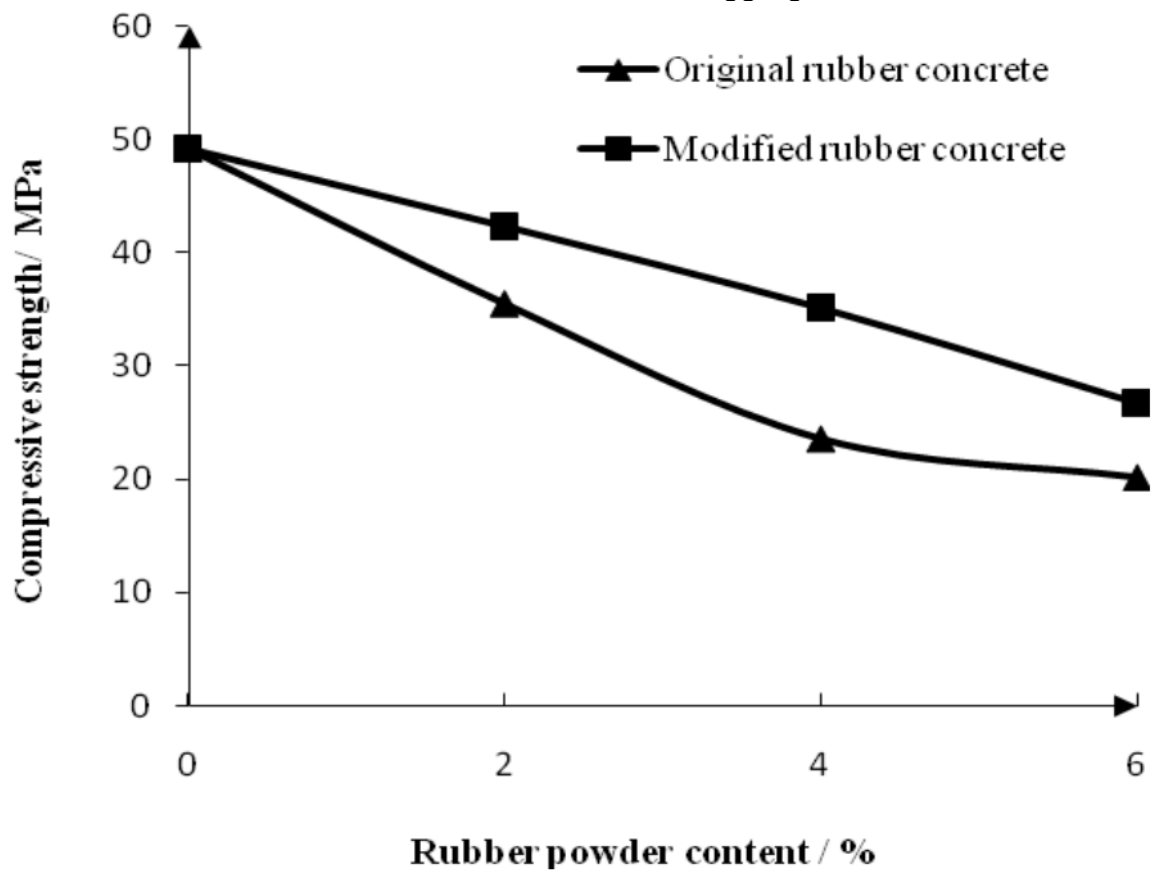
Source: (OSSOLA; WOJCIK, 2014)

2.9.5. KMnO_4 surface treatment

KMnO_4 surface treatment is an effective process to enhance the polarity of GTR (HE et al., 2016; KASHANI et al., 2018; YOUSSEF et al., 2019). The hydrophilicity

ensured to the rubber by this method promotes the adhesion between GTR particle and cement paste, which, subsequently, promotes the mechanical properties of rubberized cementitious composites, as seen in Figure 23.

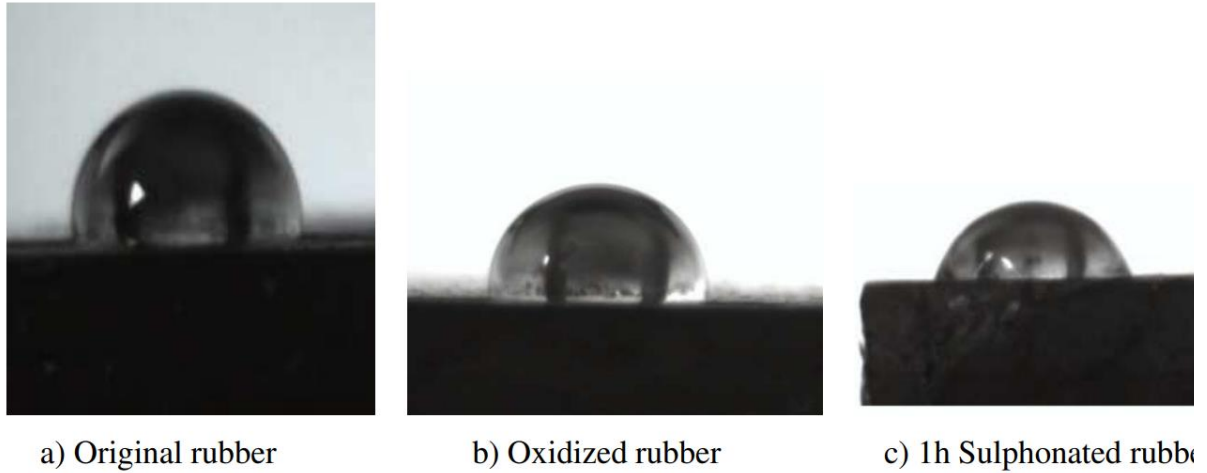
Figure 23 – Compressive strength results of concrete specimens using KMnO_4 surface treatment to the GTR aggregate



Source: (HE et al., 2016)

This method promotes the oxidation of GTR and associated to the NaHSO_3 , sulphonates rubber particle, through the creation of a large number of sulfonate, hydroxyl and carbonyl groups on the rubber surface, which reduces the contact angle with water (HE et al., 2016), as shown in Figure 24.

Figure 24 – Contact angle testing situations before and after modification of rubber



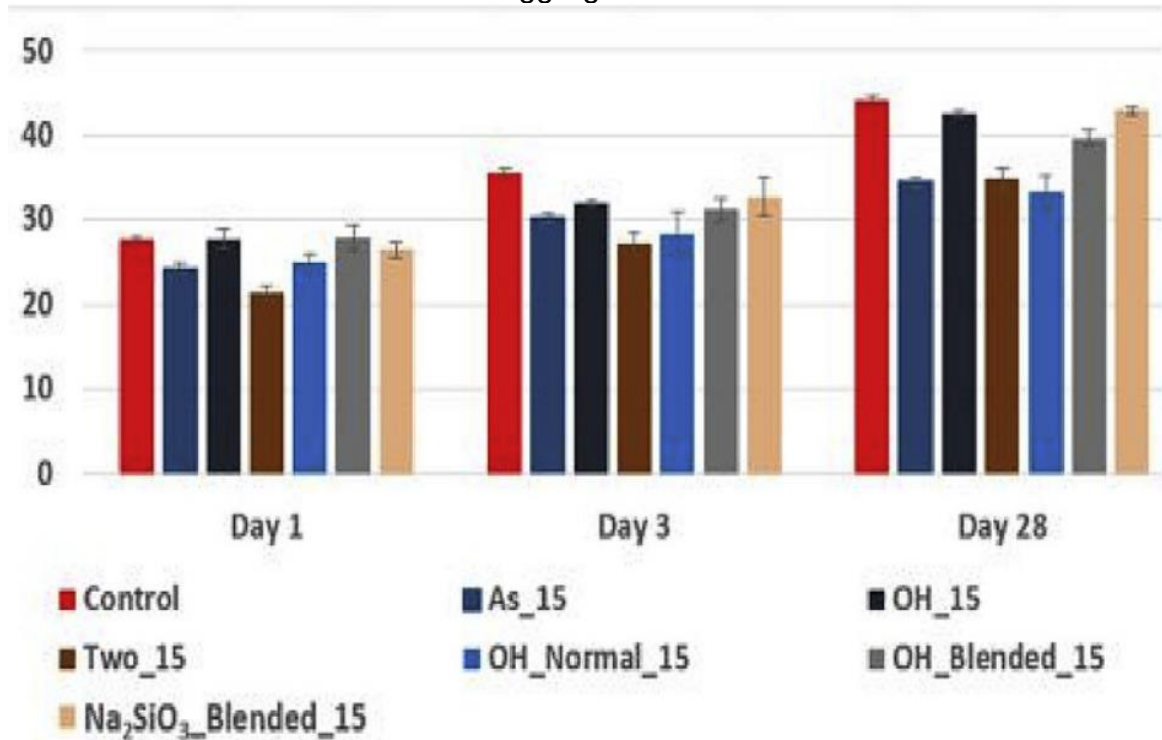
Source: (HE et al., 2016)

2.9.6. Cementitious material pre-coating

Previous rubber surface treatment using cementitious materials has many advantages, which ensures a hydrophilic surface to the aggregate and enhances cement hydration on ITZ (GUO et al., 2017; KASHANI et al., 2018; PHAM; TOUMI; TURATSINZE, 2018a, 2018b, 2019; RAFFOUL et al., 2016; ZHANG; POON, 2018).

A study performed combining pre-coating to chemical treatments showed that pre-coated samples had minimum variation of mechanical properties when compared to the reference sample, as shows Figure 25 (GUO et al., 2017). This research applied Na_2SiO_3 and NaOH to treat the rubber aggregate, then the pre-coating was applied using cement, water and silica fume on w/c ration of 0,35, and rubber/cement ration of 0,60.

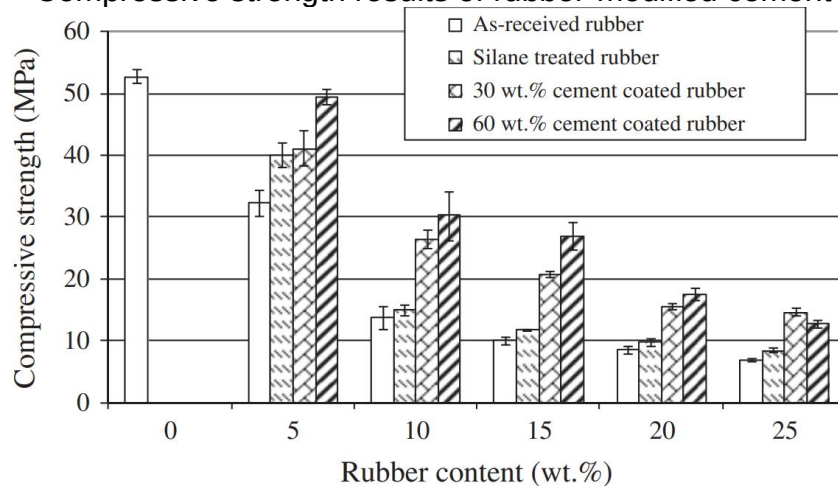
Figure 25 – Compressive strength of concrete samples with cement pre-coated rubber aggregates addition.



Source: (GUO et al., 2017)

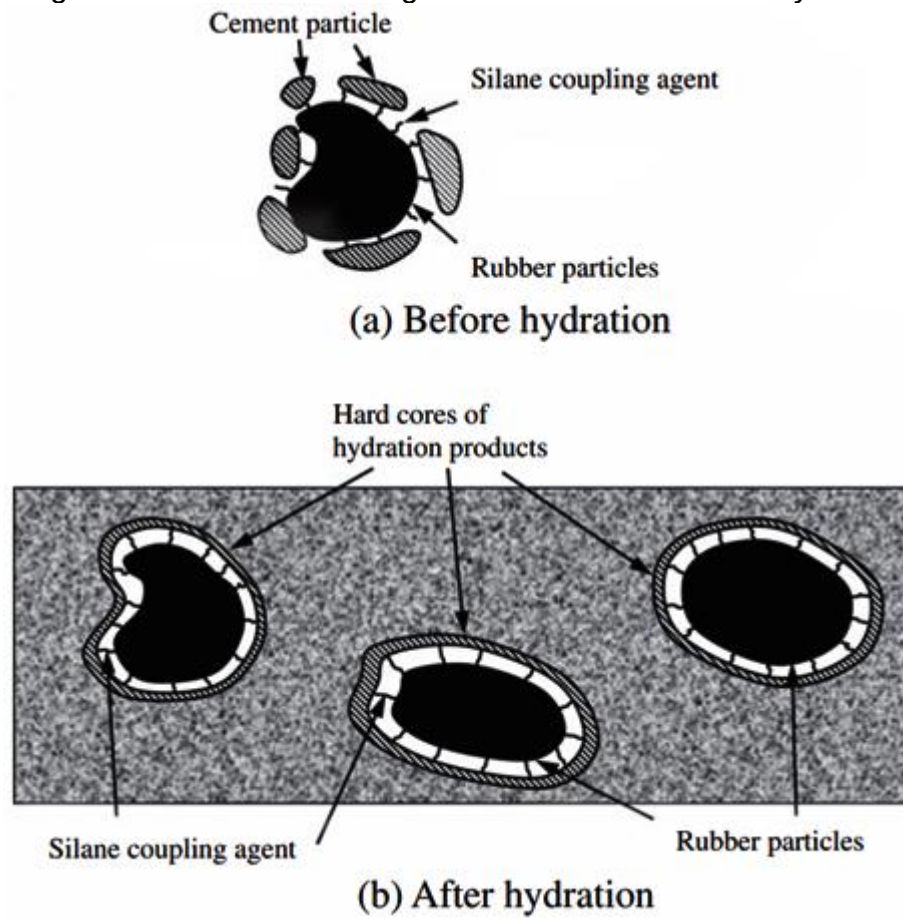
Huang et al. (2013) combined silane couple agent treatment to pre-coating and the compressive strength results were enhanced (see Figure 26). The reason for that is the hard shell created by the cement hydration, which increased the compatibility in stiffness between rubber and cement paste, as shows Figure 27 (HUANG; SHU; CAO, 2013).

Figure 26 – Compressive strength results of rubber-modified cement composites



Source: (HUANG; SHU; CAO, 2013)

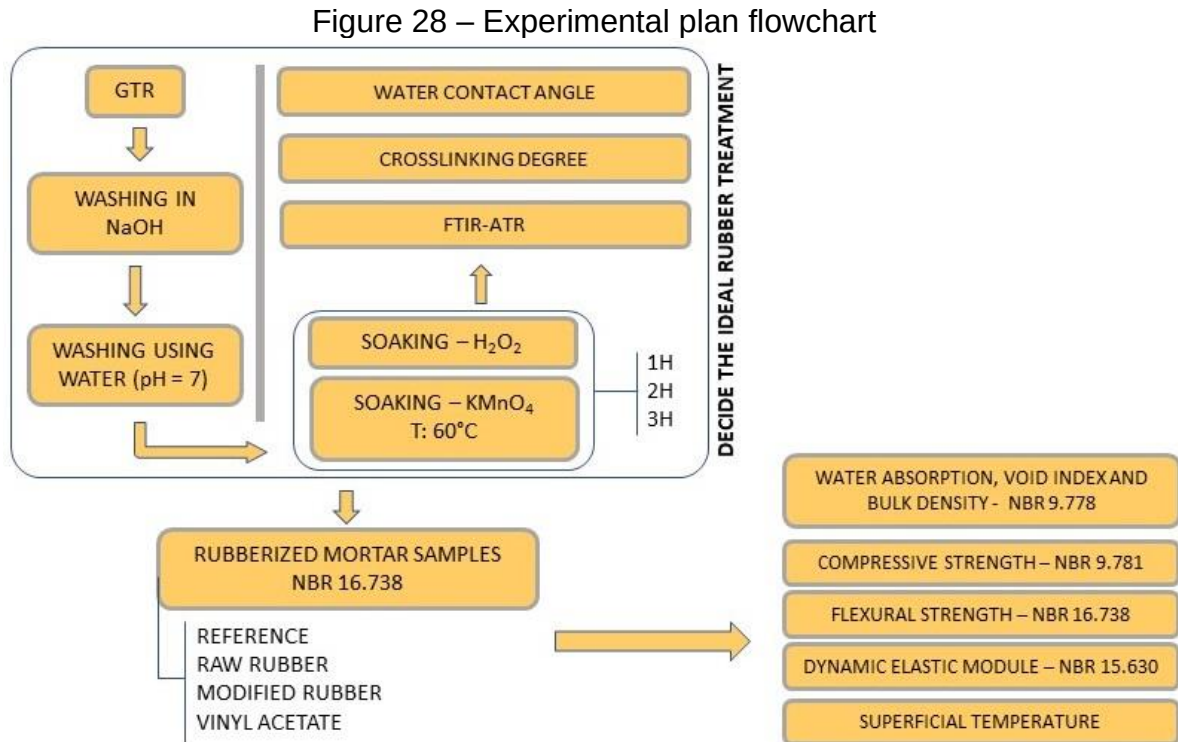
Figure 27 – Cement coating before and after cement hydration



Source: (HUANG; SHU; CAO, 2013)

3. MATERIALS AND METHODS

Figure 28 illustrates the flowchart of the entire experimental process, which was devised in conjunction with the research. The initial stage involved the treatment and characterization of Ground Tire Rubber (GTR) using two distinct chemical oxidizing agents to oxidize the rubber surface. The rubber surface was first washed with NaOH, followed by immersion in solutions of hydrogen peroxide (H_2O_2) and potassium permanganate ($KMnO_4$), separately. Subsequently, the treatment that resulted in greater hydrophilicity of the rubber particles was identified as the optimal treatment method to prepare the rubberized mortars.



Source: (Author, 2022)

3.1. Materials

The present research used to prepare the rubberized mortar specimens the following materials: Portland cement CP-V; natural sand; deionized water; GTR of 120 mesh of particle size to partially replace the sand as fine aggregate; superplasticizer; vinyl acetate emulsion; sodium hydroxide (NaOH); potassium permanganate

(KMnO₄); sulfuric acid (H₂SO₄) and hydrogen peroxide (H₂O₄). Table 2 shows the chemical composition of Portland cement CP-V, which has a specific mass of 3,14 g/cm³. Table 3 shows the chemical composition of natural sand, fine aggregate, which has a specific mass of 2,667 g/cm³.

Table 4 shows the chemical composition of 120 mesh GTR, to partially replace the fine aggregate, which has a specific mass of 1,18 g/cm³.

Table 2 – Portland cement CP-V chemical composition

Substance	Amount (%)
CaO	70,45
SiO	14,63
Al ₂ O ₃	4,90
SO ₃	4,21
Fe ₂ O ₃	3,84
K ₂ O	1,05
SrO	0,44

Source: (Cia. Nacional de Cimento, 2023)

Table 3 – Natural sand chemical composition

Substance	Amount (%)
SiO ₂	94,83
SO ₃	2,31
TiO ₂	1,48
CaO	0,96
Fe ₂ O ₃	0,38
Sm ₂ O ₃	0,15
ZnO	0,03

Source: (Author, 2022)

Table 4 – GTR chemical composition

Substance	Amount (%)
SO ₃	35,94
ZnO	33,51
SiO ₂	20,98
CaO	3,04
Br	1,67
P ₂ O ₅	1,66
Fe ₂ O ₃	1,57

Source: (Author, 2022)

3.2. Methods

3.2.1. Immersion of GTR in KMnO_4 and H_2O_2

This research applied two different GTR surface treatment on samples to define the technique which will be used to prepare the rubberized mortars (see Figure 29), and both techniques used immersion as condition to turn the rubber particle more hydrophilic: KMnO_4 and H_2O_2 .

Figure 29 – Samples of treated GTR: (A) H_2O_2 ; (B) KMnO_4 .



Source: (Author, 2022)

Washing in NaOH:

Before applying the treatment, 60g of GTR were soaked in an aqueous solution of NaOH (concentration of 10%) for 40 minutes, then they were filtered using deionized water until the pH became neutral, in order to eliminate whole solution from surface particles, avoiding its influence on results (see Figure 30).

Figure 30 – Filter system



Source: (Author, 2022)

Immersion in KMnO_4 :

The methodology described by He et al. (2016) to perform the KMnO_4 rubber surface treatment was adopted in this work. After soaking the rubber in NaOH , 10g of GTR were immersed in KMnO_4 solution for 1h, 2h and 3h at 60°C . (GTR_ KMnO_4 _1H, GTR_ KMnO_4 _2H and GTR_ KMnO_4 _3H), where an aqueous solution of KMnO_4 5% was prepared with the addition of a test tube of sulphuric acid (HE et al., 2016) to keep the p^{H} acidic to guarantee the treatment effect. The samples were dried in an oven at 60°C for 24h.

Immersion in H_2O_2 :

GTR samples were divided on three samples of approximately 10g, and then they were immersed in an aqueous solution of H_2O_2 (concentration of 5%) for three different times at room temperature: 1h, 2h and 3h (GTR_ H_2O_2 _1H, GTR_ H_2O_2 _2H and GTR_ H_2O_2 _3H).

The optimal treatment to enhance the GTR hydrophilicity and its use in mortars was defined based on the water contact angle, crosslinking degree and FTIR results.

3.2.2. Mix design, casting and curing

The prismatic mortars and the boards specimens were designed using the proportion ratio of 1: 3: 0,52: 0,007 (cement: fine aggregate: water: superplasticizer), and the fine aggregate ratio varies as the GTR replaces it by 10%, 15% and 20%. Some mixtures added a portion of vinyl acetate (VA) emulsion with the commercial name Sika® Chapisco Plus, using the proportion of 1: 0,01 (Cement: Sika® Chapisco Plus), following the manufacturer recommendations. The addition of VA emulsion in some mixtures is to investigate its influence on the studied properties with and without the chemical treatment caused by the oxidizing agents.

The prismatic specimens were molded using molds of 40 mm x 40 mm x 160 mm (width, height and length), according to NBR 16.738 standard. The present study tested at least three specimens for each characterization technique. Table 5 shows the mixture codes and replacement percentages of fine aggregate by GTR for each mixing proportion.

Table 5 – Specimens codes and components

Mix Code	Surface treatment	Additive	Amount of GTR (wt.%)
REF	-	-	0
GTR_10%	-	-	10
GTR_15%	-	-	15
GTR_20%	-	-	20
KMnO ₄ _10%	KMnO ₄	-	10
KMnO ₄ _15%	KMnO ₄	-	15
KMnO ₄ _20%	KMnO ₄	-	20
VA_REF	-	Vinyl acetate	0
VA_10%	-	Vinyl acetate	10
VA_15%	-	Vinyl acetate	15
VA_20%	-	Vinyl acetate	20
KMnO ₄ +VA_10%	KMnO ₄	Vinyl acetate	10
KMnO ₄ +VA_15%	KMnO ₄	Vinyl acetate	15
KMnO ₄ +VA_20%	KMnO ₄	Vinyl acetate	20

Source: author (2022)

The board specimens were molded using molds of 500 mm x 500 mm x 26 mm, according to an adaptation of NBR 16.738 standard. The present study used 3

specimens to the surface temperature measuring. This test was applied only on the floor boards of control and with 10 and 15% of both modified and non-modified GTR, as shown in Table 6.

Table 6 – Samples components combinations

Mix Code	Surface treatment	Amount of GTR (wt.%)
F-REF	-	0
F-GTR_10%	-	10
F-GTR_15%	-	15
F-KMnO ₄ _10%	KMnO ₄	10
F-KMnO ₄ _15%	KMnO ₄	15

Source: author (2022)

First, the dry materials were mixed for 1 minute, then they were slowly added to the liquid materials (water and superplasticizer), and all components were mixed for 1,5 minutes. The mortar then rested for 1,5 minutes, and then the materials were mixed for more 1,5 minutes. The unmolding was performed two days later, and they were cured using water immersion for one week, then the specimens were placed in air environment until completing 28 days since the mixing process.

After mixing the samples using an additive of Vinyl Acetate emulsion (VA), an excess of incorporated air was observed in the fresh state mixture, though an anti-foaming additive at a ratio of 0.1% of the water mass was added to VA group. The formation of foam when vinyl acetate is mixed within the mixture may occur due to the presence of some surfactants or foaming agents in the emulsion composition. Surfactants are compounds that possess a special molecular structure, with a hydrophilic part (affinity for water) and a lipophilic part (affinity for oily substances).

3.2.3. Characterization Methodology

3.2.3.1. Water contact angle

The water contact angle test was performed to evaluate the hydrophilicity of GTR samples according to the immersion time and type of chemical surface treatment. The test measures the angle of a drop of water on a specified surface. The results of

water contact angle test allow to analyze the studied surface polarity and its affinity with water.

The contact angle was measured using the equipment drop shape analyzer Krüss, model DSA 100 (see Figure 31). The samples were spread on a vitreous surface in order to form a thin and uniform layer of rubber.

Deionized water was used as a solvent in this procedure because it is a known polar substance. The greater the angle of the water drop in relation to the surface, the greater the difference in polarity between them, that is, the surface has low polarity (hydrophobic material).

Figure 31 – Drop shape analyzer Krüss model DSA 100



Source: (Author, 2022)

3.2.3.2. Crosslinking Degree

A tightly closed filter paper with approximately 20 mg of rubber sample was submitted to an evaporating-condensation system for 48 h at the boiling temperature of toluene, used as the solvent (see Figure 32). The cross-link degree, related to the insoluble residue, was calculated as the ratio between the weights of the sample after and before being soaked in toluene during the evaporation process. Each sample was tested three times.

Figure 32 – Crosslinking degree test



Source: (Author, 2022)

The possibility of filter paper solvation in the solvent was considered, thus it was necessary to eliminate the solved portion of the filter paper from the entire weight of the sample plus the filter paper after the test, following Equations (1) and (2):

$$CD\% = \frac{W_t - \{W_b - (W_b \cdot Lfp\%)\}}{W_s} \times 100 \quad (1)$$

$$Lfp\% = \left(1 - \frac{B_a}{B_b}\right) \times 100 \quad (2)$$

where CD is the cross-link degree (%), W_t is the real total weight of sample with filter paper after the test (mg), W_b is the weight of sample and filter paper before the test (mg), W_s is the weight of the sample (mg), L_{fp} is the filter paper weight loss (mg) due to the contact with solvent (Applicable for the same type of filter paper), B_a is the blank filter paper weight after the test (mg), and B_b is the blank filter paper initial weight (mg).

3.2.3.3. Fourier transform infrared spectroscopy (FTIR-ATR)

The infrared spectra were obtained using a Varian Spectrophotometer 3100 FTIR Excalibur Series (Japan). The treated tire rubber samples were macerated with potassium bromide (KBr) to form pellets, where 1 mg of each sample was weighed and mixed with 300 mg of KBr. Subsequently, the FTIR spectrum of the sample was recorded at room temperature with a resolution of 4 cm⁻¹, 64 accumulated scans, and a wavenumber range of 4000 to 800 cm⁻¹, using the transmittance mode. Quantification of the bands was performed by calculating the difference in area between each group of similar bands. The process was conducted in duplicate.

3.2.4. Water absorption, void ratio and bulk density

The water absorption, void ratio and bulk density tests were performed, using specimens of 40 mm x 40 mm x 40 mm, at 28 days of curing, according to the NBR 9.778. The specimens were prepared and cured as described in item 3.2.2, and then were submitted to a kiln at 40°C, where they remained for 5 days to acquire mass constancy. After this period, the mass of each specimen was measured using analytical balance and recorded as a constant dry mass (m_d). Subsequently, they were immersed in water for 72 hours to reach saturation. Once saturated, the specimens were immersed in a glass beaker of water with 2 liters of volume capacity, and then the baker was replaced on a hot plate. The water temperature was elevated to its boiling point between 15 and 30 min. The boiling was maintained for 5 hours. After this period, the beaker was removed and cooled naturally. Then each specimen was weighed individually using a hydrostatic balance and recorded its mass as immersed mass (m_i). The next step was then drying it with paper tissues and weighing it again and the mass was then recorded as saturated surface dry mass (m_{sat}). The void ratio value (V_c), total absorption (A) and the bulk density (ρ_r) were obtained applying the following equations (3), (4) and (5), respectively:

$$V_c = \frac{m_{sat} - m_d}{m_{sat} - m_i} \cdot 100 \quad (3)$$

$$A = \frac{m_{sat}-m_d}{m_d} .100 \quad (4)$$

$$\rho_r = \frac{m_d}{m_d - m_i} \quad (5)$$

3.2.5. Compressive strength

The compressive strength test was performed, using specimens of 40 mm x 40 mm x 40 mm, at 28 days of curing. The test used a Kratos hydraulic press with 200 kN load capacity. The load rate was 720 N/s, according to the NBR 16.738.

3.2.6. Flexural strength

The flexural strength test was performed using 3 samples of 40 mm x 40 mm x 160 mm, at 28 days age. The test used a Kratos hydraulic press with 200 kN load capacity. The load rate was 50 N/s according to the NBR 12.142 standard.

3.2.7. Dynamic elastic modulus

The Dynamic Elastic Modulus test was performed on three specimens of 40 mm x 40 mm x 160 mm at 28 days of age, following the guidelines outlined in NBR 15630 (2008). The wave propagation velocity is calculated using equation (6):

$$V = \frac{L}{t} \quad (6)$$

Where:

- V: ultrasonic wave propagation speed, expressed in millimeters per microsecond (mm/μs).
- L: distance between the coupling point of the transducers (mm).
- t: time recorded by the digital display (μs).

The dynamic modulus of elasticity can be calculated according to equation (7):

$$E_d = V^2 \rho \frac{(1 + \mu)(1 - 2\mu)}{1 - \mu} \quad (7)$$

Where:

- E_d : dynamic elastic modulus, expressed in megapascals (MPa).
- ρ : bulk density of the specimen, expressed in kilograms per cubic meter.
- μ : Poisson's ratio, in this equation, the value of 0.2 is adopted.
- V : ultrasonic wave propagation speed, expressed in millimeters per microsecond (mm/ μ s).
- L : distance between the coupling point of the transducers (mm).
- t : time recorded by the digital display (μ s).

3.2.8. Superficial temperature test

The thermal properties of the floor boards are intended to be studied by monitoring the surface temperature changes on their surface while exposed to sun. In Figure 33, the prepared floor boards were exposed to solar radiation in an outdoor environment, and pairs of thermocouples were installed on the surface to measure the surface temperatures of the specimens.

Monitoring the surface temperature was performed applying two sets of action; the first set was during three days every 5 minutes monitoring only the exposed side to the solar radiation during day and night. For data optimization, the author explored the thermal response of the thermocouples during 6 hours only from 09 am to 03 pm. The floor boards were coded as: REF, GTR_10%, GTR_15%, KMnO4_10%, and KMnO4_15%. This test was applied only on the floor boards of control and with 10 and 15% of both modified and non-modified GTR; and the second set was during eight days every 5 minutes monitoring the heat flux transferred from the exposed side to the other one, represented by the difference of the temperatures of both sides. This second set was intended to explore how the boards act with and without rubber transferring heat from side to another.

Additionally, the values of the climatic conditions at the site were registered, simultaneously, through a meteorological station installed at the Technological Research Institute (IPT) in São Paulo/SP.

Figure 33 – Floor boards with thermocouple poles installed for the surface temperature test.



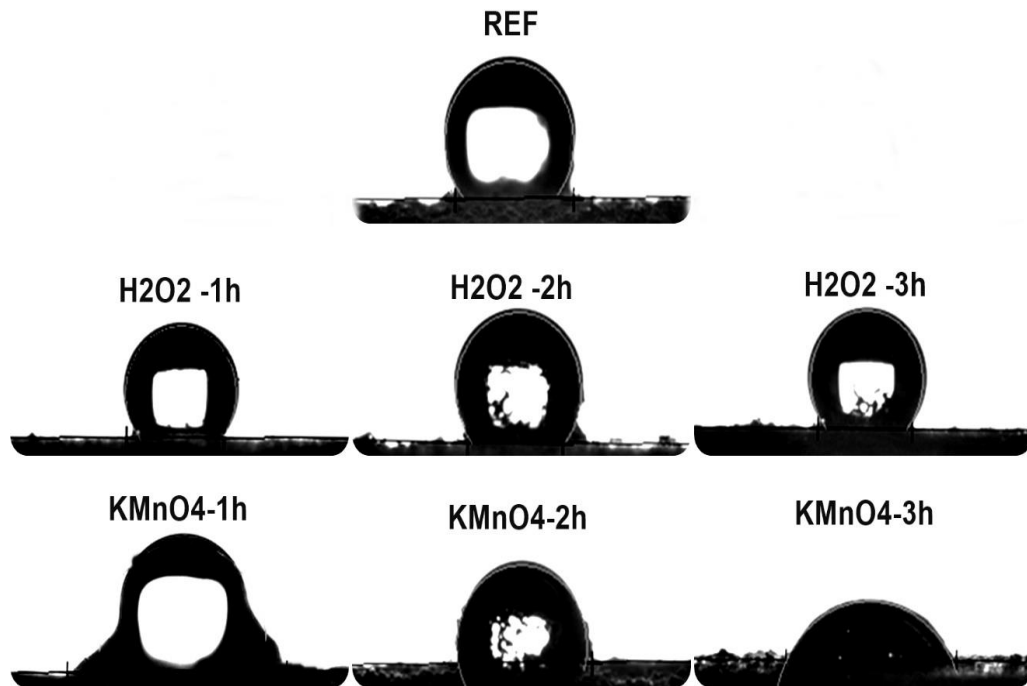
Source: (Author, 2023)

4. RESULTS AND DISCUSSIONS

4.1. Contact angle

The contact angle test measures the affinity between water and GTR particles and the present study used this assay to define the optimal rubber surface treatment to apply the treated GTR in the mortar mixtures. Figure 34 shows the contact angle images to the GTR surface before and after the treatment as a function of soaking time.

Figure 34 – Contact angle variation of GTR as a function of soaking time with different surface treatments.



Source: (Author, 2022)

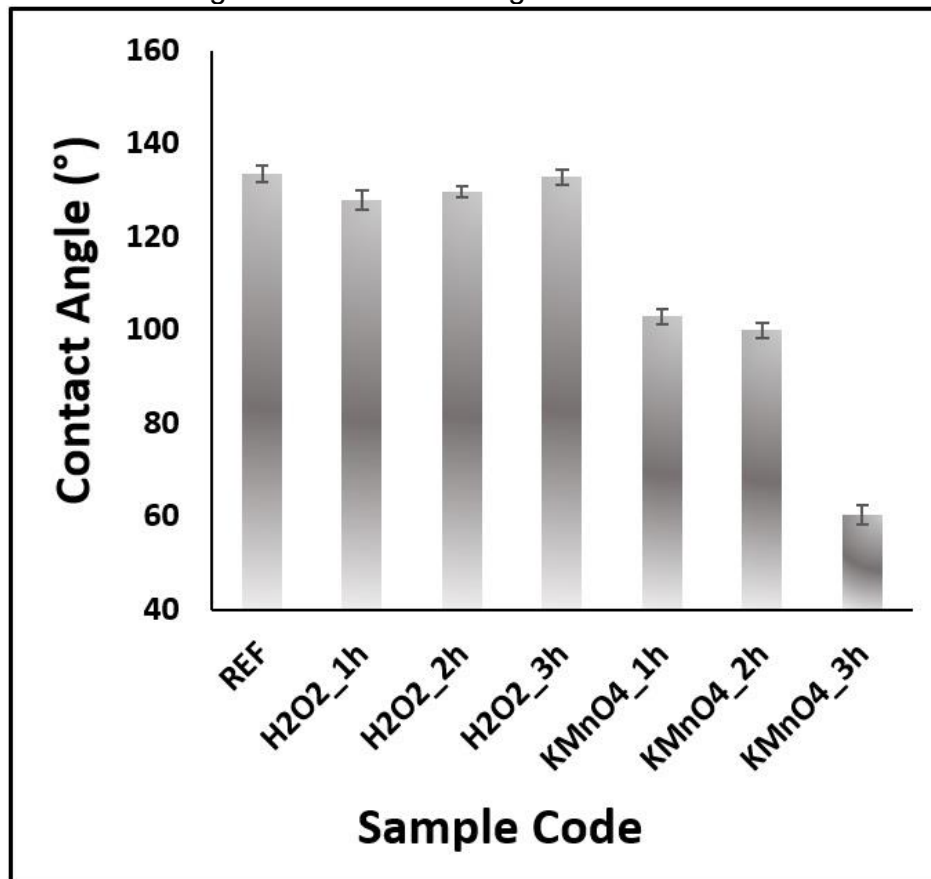
The presented results in

Figure 35, demonstrate that KMnO4 GTR surface treatment is the most efficient in terms of introducing polar groups on the surface of the GTR, and the contact angle is reduced as a function of immersion time. The control sample presented a contact angle of 136° , which reflects how hydrophobic is, meanwhile, the GTR samples treated using H2O2 did not present significant variations of the contact angle when compared to the reference sample. On the other hand, it was obvious to notice the influence of

the KMnO₄ solution on the surface of the GTR, as the contact angle could be reduced to 60°, which means a reduction of 56%, indicating a clear hydrophobic character absence of the surface of the GTR soaked for 3 hours into KMnO₄. This finding corroborates the FTIR spectra where new polar functional groups were detected on the KMnO₄ treated GTR samples.

Therefore, the GTR samples treated with KMnO₄ for 3 hours present feasibility to be applied in cementitious matrices due to presenting more hydrophilic character, while the other treatments did not change the hydrophobic nature of GTR. The present study used the KMnO₄ rubber surface treatment as the optimal treatment of the GTR to partially replace the sand in mortar mixtures.

Figure 35 – Contact angle of GTR treated

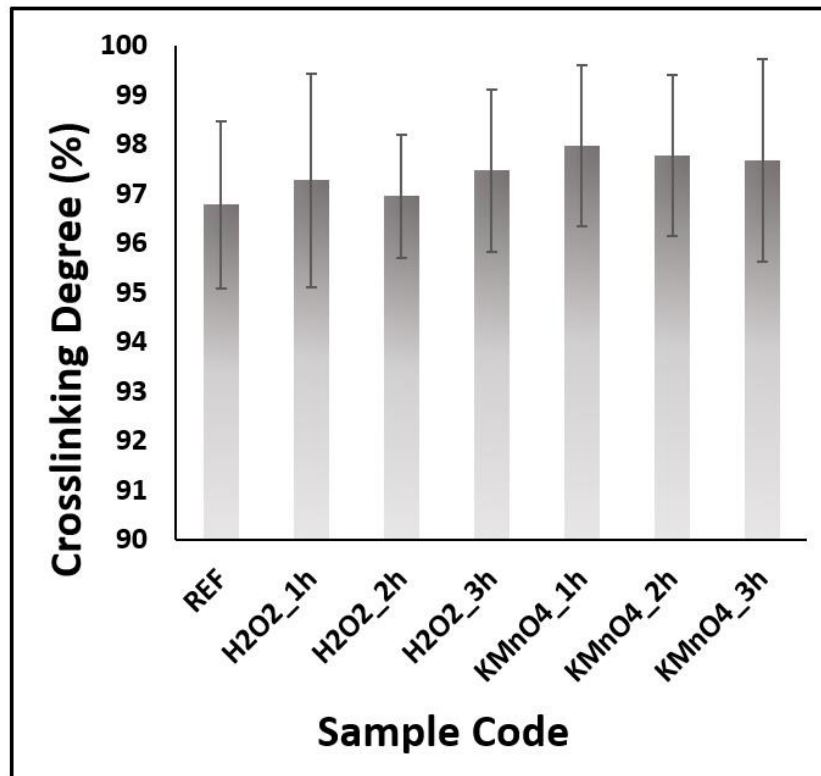


Source: (Author, 2023)

4.2. Crosslinks

Figure 38 shows the change in the crosslinking degree of ground tire rubber (GTR) before and after the chemical treatments. It can be observed that all the GTR samples present high values of crosslinking regardless of the applied treatment agent or soaking time. The control GTR sample REF kept 96.79 % of crosslinking, while the GTR samples treated by H_2O_2 kept 97.29, 96.96 and 97.47 % of crosslinking and KMnO_4 kept 97.98, 97.78, and 97.69 % of crosslinking after a contact of 1, 2 and 3 hours, respectively. Applying ANOVA test, it was revealed that all the results averages presented very close distribution considering the error bars (see Figure 36) and there is no significant difference between them. One can conclude that the applied treatments had no significant influence on the crosslinking degree of the GTR samples and the carbon-sulfur bonds were intact, according to the presented findings.

Figure 36 – Change in the crosslinking degree of ground tire rubber (GTR) before and after the chemical treatments



Source: (Author, 2023)

4.3. FTIR

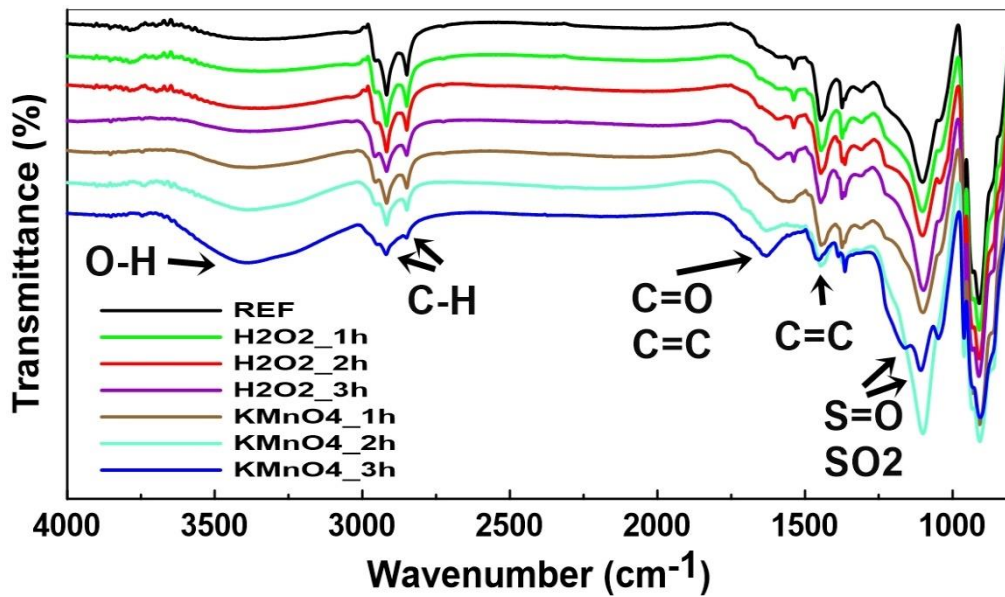
Figure 36 shows the FTIR spectra of the GTR samples before and after the chemical treatment. All vibration modes of the FTIR spectra are represented in Table 7. In the rubber spectra, presented in Figure 36, it is possible to observe some important characteristic bands: a wide characteristic band at 3350 cm^{-1} , attributed to the OH elongation; a doublet of characteristic bands at 2916 and 2848 cm^{-1} , related to the elongation of the C-H bond in CH_2 and CH_3 groups; the characteristic band at 1637 cm^{-1} , designated as the C=O conjugate with the C=C elongation; 3 characteristic bands at 1537 , 1493 and 1455 cm^{-1} , respectively, attributed to asymmetric and symmetric C=C elongation in the aromatic ring skeleton; the characteristic band at 1085 cm^{-1} , designated as S=O and SO^2 -conjugated elongation; and the characteristic band at 808 cm^{-1} , designated as C-H curvature of the out-of-plane ring. The characteristic bands at 2916 , 2848 , 1537 , 1493 , and 1455 cm^{-1} , representing the main GTR rubber bands (ABOELKHEIR et al., 2021a; BUONERBA et al., 2014; SHAO et al., 2016; SILVERSTEIN; WEBSTER, 2000; WANG et al., 2015).

The characteristic band at 3350 cm^{-1} , attributed as the O-H elongation increased significantly by increasing the soaking time in KMnO_4 which indicates higher polarity of the GTR surface due to the oxidation process if compared to the control sample REF. It can be noticed also the increasing appearance of the characteristic band, at 1637 cm^{-1} , assigned as the C=O conjugated with C=C stretching which also may indicated higher polarity due to the carbonyl group contribution. The same reasoning can be applied by analyzing the S=O and SO_2 conjugated stretching occurred at 1035 cm^{-1} , which also may indicate higher polarity of the GTR surface if compared to the control sample. These findings corroborate the results of the water contact angle test, where the KMnO_4 treated GTR samples presented the lowest angles with a reduction of 56 %, if compared to the control sample.

Table 7 – Index of vibration modes of ground tire rubber (GTR) FTIR spectra before and after the chemical treatments.

Characteristic Band (cm ⁻¹)	Vibration Modes
3350	O-H stretching
2916	C-H stretching bond in CH ₂ and CH ₃ groups
2848	
1637	C=O & C=C conjugated stretching
1537	C=C asymmetric and symmetric stretching in the aromatic ring skeleton
1493	
1455	
1085	S=O & SO ₂ conjugated stretching associated with Si-O-Si stretching
808	C-H out-of-plane ring

Figure 37 – Index of vibration modes of ground tire rubber (GTR) FTIR spectra before and after the chemical treatments



Source: (Author, 2023)

4.4. Water absorption, void ratio and bulk density

4.4.1. Water absorption

Table 8 presents the values of the total water absorption test of the samples after 28 days of curing. The presented results provide the influence of the GTR on the permeability of the specimens.

The analysis conducted in Figure 38 underscores notable insights concerning the water absorption characteristics of different samples. Specifically, when comparing reference samples with those subjected to treatments involving both treated and untreated rubber compounds, and notably in the absence of GTR, the variations in water absorption values appear to be of marginal significance.

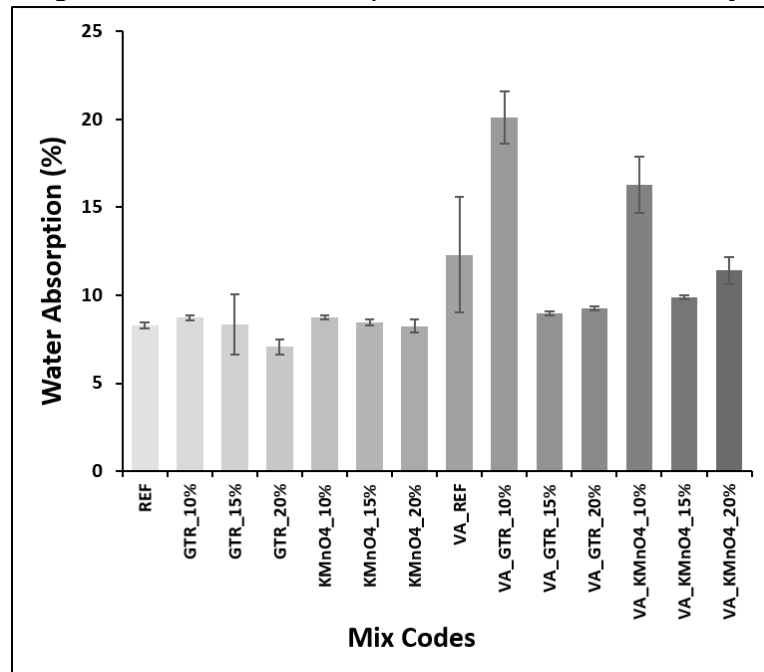
However, a distinct trend emerges when samples incorporating VA are examined. In such instances, a substantial increase in permeability is observed. This shift is attributed to the creation of bubbles during the blending process, a consequence of the interaction between the emulsion and cement hydration products. These bubbles, carrying entrapped air, persist within the mortar both during the mixing stage and the subsequent curing process.

The repercussions of this phenomenon manifest significantly in water absorption behavior. The presence of a higher volume of entrapped air and the corresponding increase in porosity within the specimens result in a pronounced elevation in water absorption capacity. This stands in stark contrast to the control specimens, further highlighting the influential role of VA in altering the material's permeability and porosity.

Table 8 – Water absorption test results at 28 days

Mix Code	Water Absorption (%)	Standard Deviation (%)	CoV (%)
REF	8.31	0.16	1.98
GTR_10%	8.73	0.13	1.48
GTR_15%	8.36	1.71	20.48
GTR_20%	7.08	0.42	5.97
KMnO4_10%	8.74	0.13	1.49
KMnO4_15%	8.46	0.15	1.83
KMnO4_20%	8.26	0.35	4.27
VA_REF	12.31	3.29	26.72
VA_GTR_10%	20.08	1.49	7.41
VA_GTR_15%	8.97	0.10	1.06
VA_GTR_20%	9.27	0.12	1.27
VA_KMnO4_10%	16.27	1.61	9.87
VA_KMnO4_15%	9.89	0.12	1.16
VA_KMnO4_20%	11.41	0.77	6.79

Figure 38 – Water absorption test results at 28 days



Source: (Author, 2023)

It is important to highlight that due to the difficulties encountered in compacting the mixture after blending the components, the associated error was increased for the samples coded VA_REF, VA_GTR_10%, VA_KMnO4_10%, and VA_KMnO4_20%.

4.4.2. Void Ratio

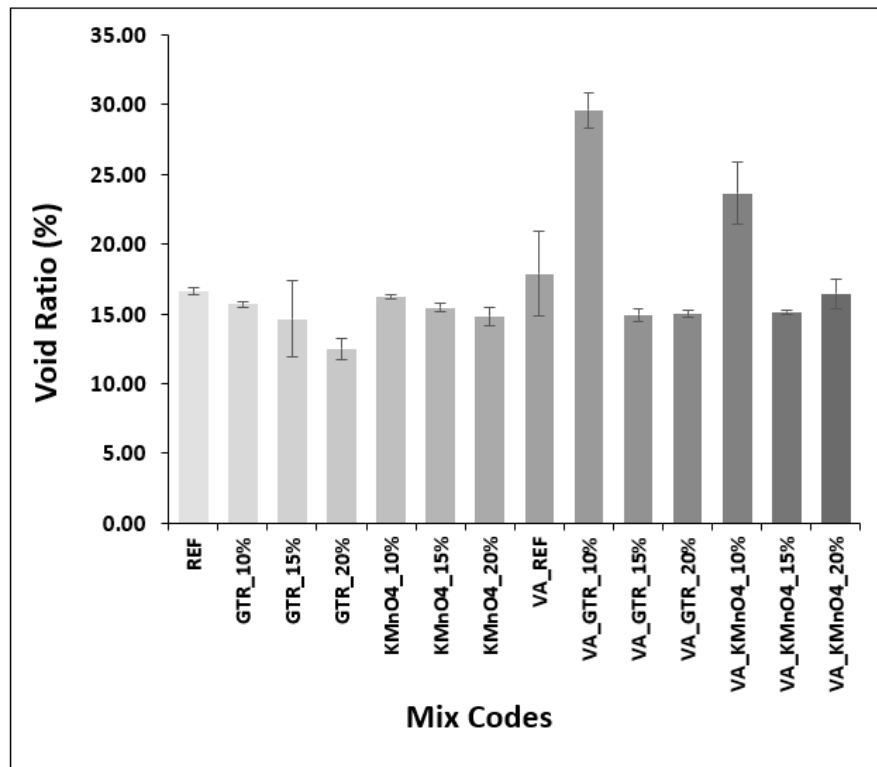
Table 9 presents the values obtained in the void index test of the samples after 28 days of curing. The results presented provide an understanding of the influence of rubber on water absorption and compaction of the specimens.

From Figure 39, it is perceptible that, in the case of samples featuring untreated rubber, the trend entails a reduction in the void index as the volumetric proportion of recycled aggregate experiences augmentation. Conspicuously, when rubber undergoes pre-treatment, the samples evince minimal fluctuations in the void index, in contradistinction to the reference sample.

Table 9 – Void ratio test results at 28 days

Mix Code	Void Ratio (%)	Standard Deviation (%)	CoV (%)
REF	16.62	0.30	1.80
GTR_10%	15.67	0.19	1.21
GTR_15%	14.62	2.73	18.67
GTR_20%	12.48	0.76	6.07
KMnO4_10%	16.21	0.15	0.93
KMnO4_15%	15.44	0.30	1.91
KMnO4_20%	14.82	0.68	4.60
VA_REF	17.89	3.08	17.19
VA_GTR_10%	29.61	1.27	4.29
VA_GTR_15%	14.91	0.45	2.99
VA_GTR_20%	14.97	0.25	1.70
VA_KMnO4_10%	23.65	2.23	9.42
VA_KMnO4_15%	15.12	0.14	0.90
VA_KMnO4_20%	16.44	1.08	6.55

Figure 39 – Void ratio test results at 28 days



Source: (Author, 2023)

Analogous to the water absorption tendencies, specimens integrated with vinyl acetate content exhibit a discernibly amplified prevalence of voids embedded within their structure. The discerned variability in the standard deviation is attributed to the

inherent challenges encountered in effectively compacting the composite mixture, which, in turn, influences the homogeneity of void distribution.

4.4.3. Bulk density

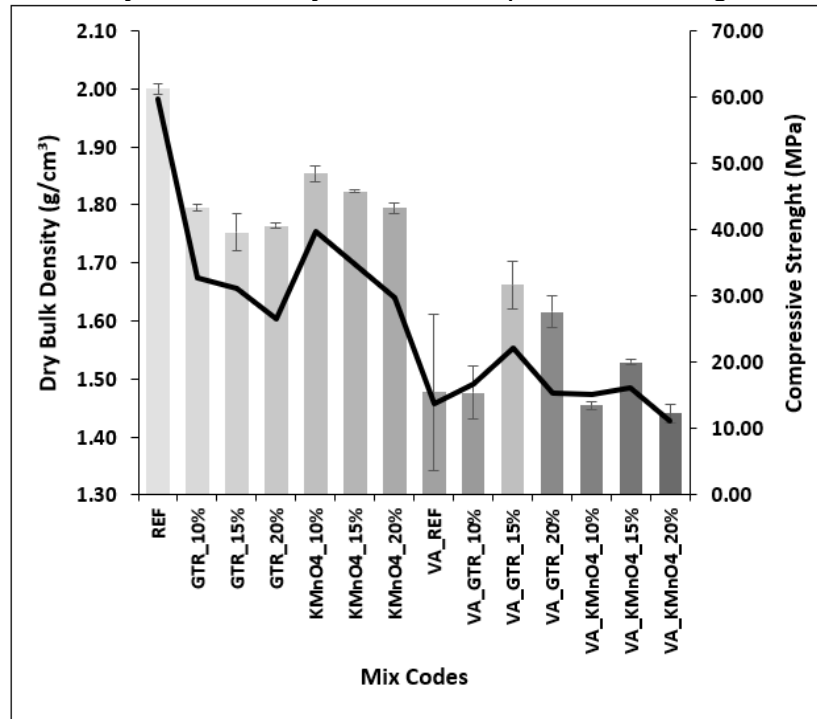
The determined values attributed to dry bulk density of the studied composites are elucidated within Table 10. These findings furnish a comprehension of the consequential impact of rubber content on the diminution of composite density.

Figure 40 shows the variation of dry bulk density as a function of the percentage of sand substitution by ground rubber, surface treatment, and addition of vinyl acetate versus the compressive strength. The specimens with VA presented less bulk density than the others. Additionally, the surface treatment of the rubber ensured higher dry bulk densities than those without treatment and those or with VA. This is due to the improved adhesion of the cement paste with the recycled aggregate, enhancing cement hydration and the formation of its products. These findings are corroborated with the compressive strength behavior of the specimens with treated GTR.

Table 10 – Dry Bulk Density at 28 Days

Mix Code	Dry Bulk Density (g/cm³)	Standard Deviation (g/cm³)	CoV (%)
REF	2.00	0.01	0.46
GTR_10%	1.80	0.01	0.29
GTR_15%	1.75	0.03	1.81
GTR_20%	1.76	0.00	0.25
KMnO4_10%	1.85	0.01	0.70
KMnO4_15%	1.82	0.00	0.16
KMnO4_20%	1.79	0.01	0.54
VA_REF	1.48	0.13	9.12
VA_GTR_10%	1.48	0.04	3.03
VA_GTR_15%	1.66	0.04	2.52
VA_GTR_20%	1.62	0.03	1.68
VA_KMnO4_10%	1.45	0.01	0.52
VA_KMnO4_15%	1.53	0.00	0.27
VA_KMnO4_20%	1.44	0.02	1.12

Figure 40 – Dry Bulk Density versus Compressive Strength at 28 Days



Source: (Author, 2023)

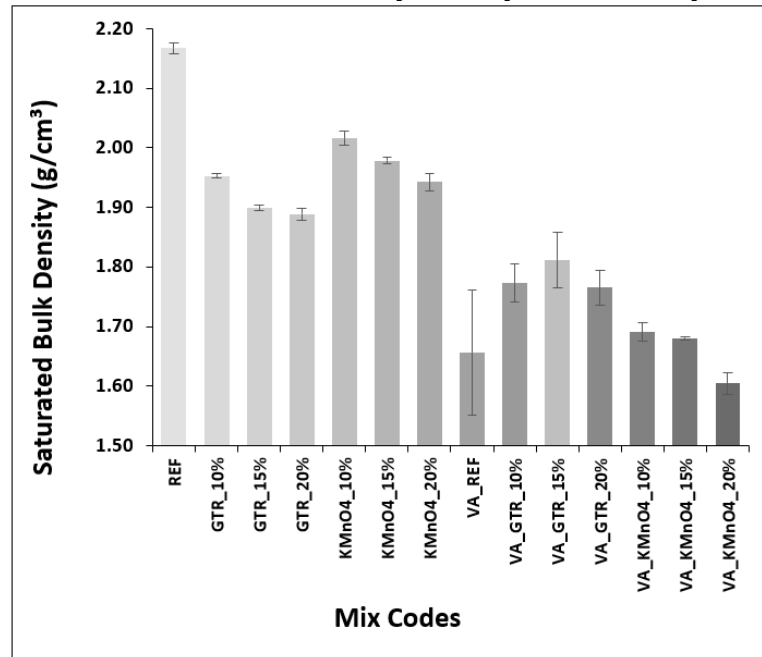
The compressive strength of the test specimens is directly proportional to the dry bulk density of the samples. This phenomenon can be explained by two factors: the greater the amount of rubber, the lower the dry bulk density, as rubber is lighter than sand, and there are more voids in the hardened mortar, which weaken the composite.

Table 11 provides a comprehensive exposition of the saturated bulk density results pertaining to the specimens under consideration. These findings are graphically depicted in Figure 41, which serves as an illustrative tool to visually encapsulate the nuances of the saturated density exhibited by the test specimens. Evidently, a discernible augmentation in the bulk density of the samples becomes apparent subsequent to their submersion in water. This discernible transformation is a direct outcome of the water absorption process undertaken by the cementitious composite. Remarkably, the specimens infused with vinyl acetate showcase a significantly amplified variance between their dry and saturated bulk densities. This particular distinction can be attributed to an augmented void ratio inherent within these samples, emanating from the intricate process of compaction hindered by the presence of entrapped air bubbles within the mixture.

Table 11 – Saturated Bulk Density at 28 Days

Mix Code	Saturated Bulk Density (g/cm ³)	Standard Deviation (g/cm ³)	CoV (%)
REF	2.17	0.01	0.41
GTR_10%	1.95	0.00	0.19
GTR_15%	1.90	0.00	0.23
GTR_20%	1.89	0.01	0.54
KMnO4_10%	2.02	0.01	0.60
KMnO4_15%	1.98	0.01	0.26
KMnO4_20%	1.94	0.01	0.77
VA_REF	1.66	0.11	6.37
VA_GTR_10%	1.77	0.03	1.81
VA_GTR_15%	1.81	0.05	2.55
VA_GTR_20%	1.77	0.03	1.64
VA_KMnO4_10%	1.69	0.02	0.92
VA_KMnO4_15%	1.68	0.00	0.16
VA_KMnO4_20%	1.60	0.02	1.14

Figure 41 – Saturated Bulk Density vs. Dry Bulk Density at 28 Days



Source: (Author, 2023)

4.5. Compressive strength

The results of the compressive strength test conducted on the prismatic specimens after 28 days of curing are presented in Table 12. Figure 42 depicts the

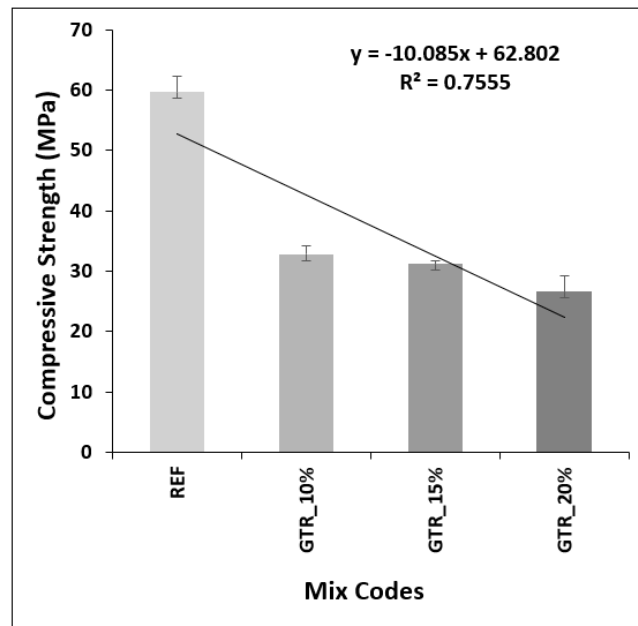
graph of the average compressive strength of the reference specimens and the specimens with untreated rubber, varying the percentage of fine aggregate replacement by the elastomer. It can be observed that there is a significant reduction in compressive strength with the addition of GTR in the mixture, which is directly proportional to the volumetric fraction of substitution.

The compressive strength of the specimens with GTR replacement at volumetric fractions of 10%, 15%, and 20% exhibited significant reductions compared to the reference sample (REF). The reductions were approximately 45%, 48%, and 55% in compressive strength, respectively. This reduction can be attributed to the hydrophobic nature of the rubber, which hinders proper hydration of the cement on the rubber's surface. Consequently, it leads to a weaker bond and increased brittleness at the interface of the transition zone.

Table 12 – Compressive strength test results at 28 days

MIX Code	Compressive Strength (MPa)	Standard Deviation (MPa)	CoV (%)
REF	59.71	2.67	4.47
GTR_10%	32.79	1.49	4.55
GTR_15%	31.25	0.54	1.74
GTR_20%	26.60	2.59	9.73
KMnO4_10%	39.73	0.79	1.99
KMnO4_15%	34.79	2.52	7.24
KMnO4_20%	29.71	1.43	4.82
VA_REF	13.75	5.47	39.79
VA_GTR_10%	16.67	0.79	4.76
VA_GTR_15%	22.17	2.25	10.17
VA_GTR_20%	15.35	3.92	25.54
VA_KMnO4_10%	15.23	0.14	0.95
VA_KMnO4_15%	16.08	2.46	15.28
VA_KMnO4_20%	11.10	0.91	8.20

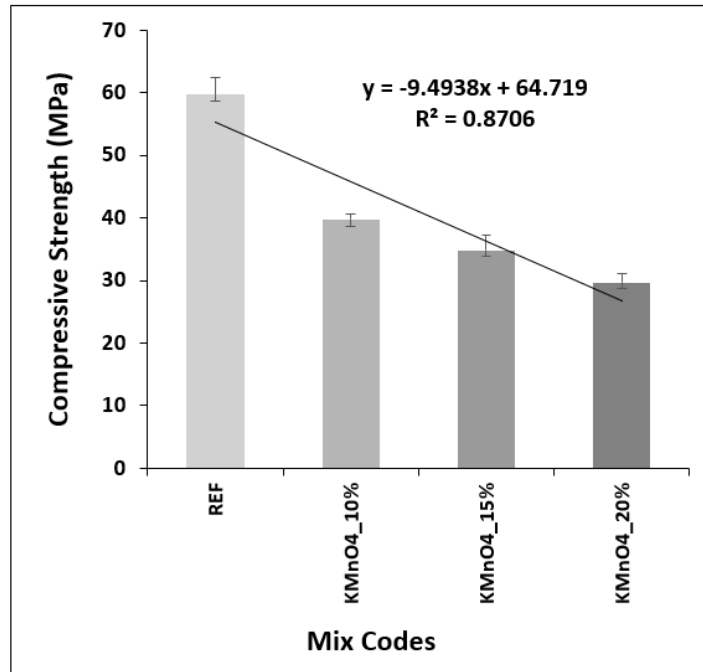
Figure 42 – Compressive strength for the reference mixtures and mixtures with untreated GTR addition.



Source: (Author, 2023)

Figure 43 illustrates the influence on compressive strength after the addition of rubber treated with potassium permanganate at 60°C for 3 hours to the mortar. The specimens with treated GTR show improvements in compressive strength performance compared to untreated samples. However, the values remain below the reference sample. The decreases in strength increase as the replacement of fine aggregate increases, ranging from 33% to 50%. It can be concluded that the optimal volumetric fraction for replacement of fine aggregate with treated rubber is 10%, with is with accordance with the literature (ABOELKHEIR et al., 2021b).

Figure 43 – Compressive strength for the reference mixtures and mixtures with KMnO₄ surface treatment.

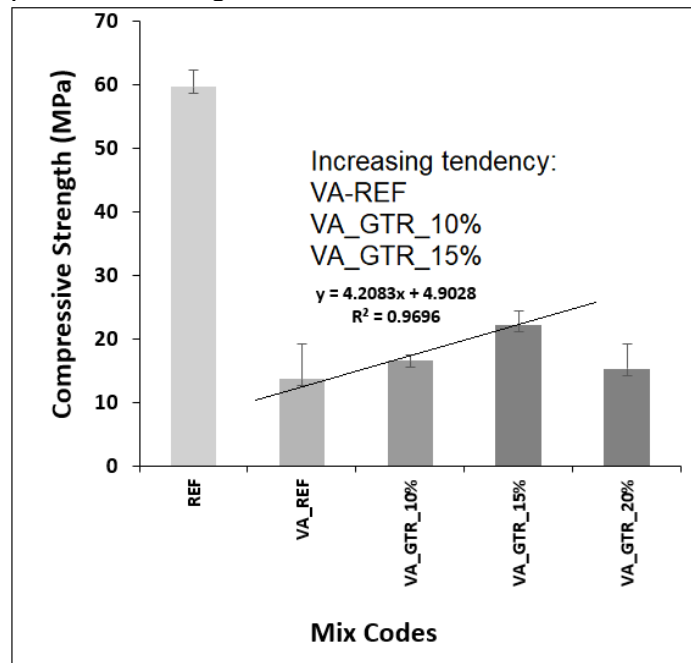


Source: (Author, 2023)

On the other hand, an increase in the compressive strength is observed in the treated samples compared to the untreated ones. For the 10% fraction, there was a 21% increase in compressive strength. This difference is attributed to the oxidation potential of potassium permanganate, which transforms the rubber from non-polar to gain polarity by introducing polar functional groups on its surface, enhancing the hydrophilicity. This facilitates water migration within the mixture and promotes hydration of the cement on the rubber's surface.

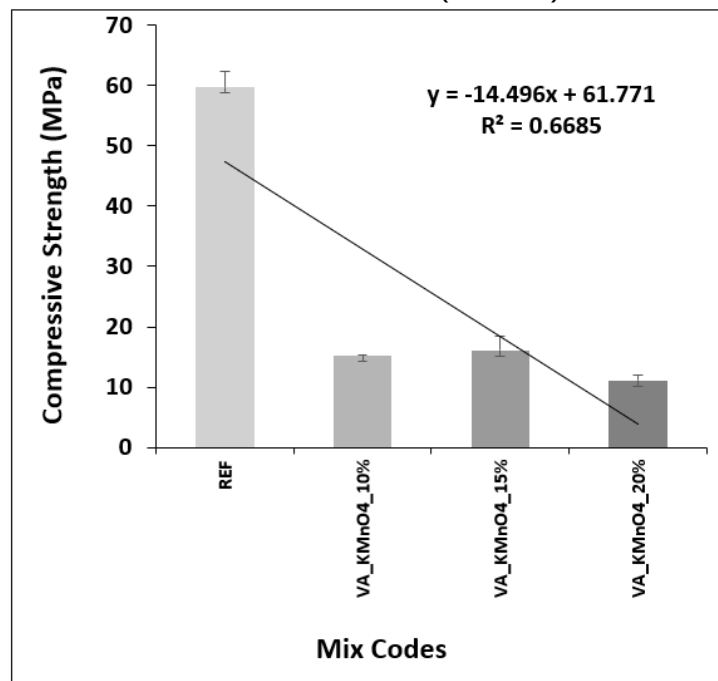
Figure 44 represents the compressive strength values of the specimens with untreated rubber and the addition of (VA). Meanwhile, Figure 45 displays the compressive strength values of the specimens with treated rubber and the addition of Vinyl Acetate (VA). Both Figures are describing the influence of VA as an admixture additive on the studied properties in the presence of treated and untreated GTR.

Figure 44 – Compressive strength for the reference mixtures and mixtures with VA



Source: (Author, 2023)

Figure 45 – Compressive strength for the reference mixtures, mixtures with VA and surface treatment (KMnO₄)



Source: (Author, 2023)

The compressive strength results of the specimens with untreated and treated rubber, both with the addition of VA, presented lower values compared to all the other studied specimens. This phenomenon can be attributed to the presence of bubbles

that formed within the fresh mixture. Despite the addition of an anti-foaming additive, bubbles continued to appear and hindered the compaction of the samples, resulting in a high void content and ultimately leading to poor mechanical properties.

Worth mentioning that the compressive strength of the specimens with VA additive showcased an increasing tendency when VA was added with 10 and 15% of GTR, which may indicate that the fine particles of GTR acted as a filler and partially reduced the void content, leading to a slight increase in compressive strength (see Figure 44).

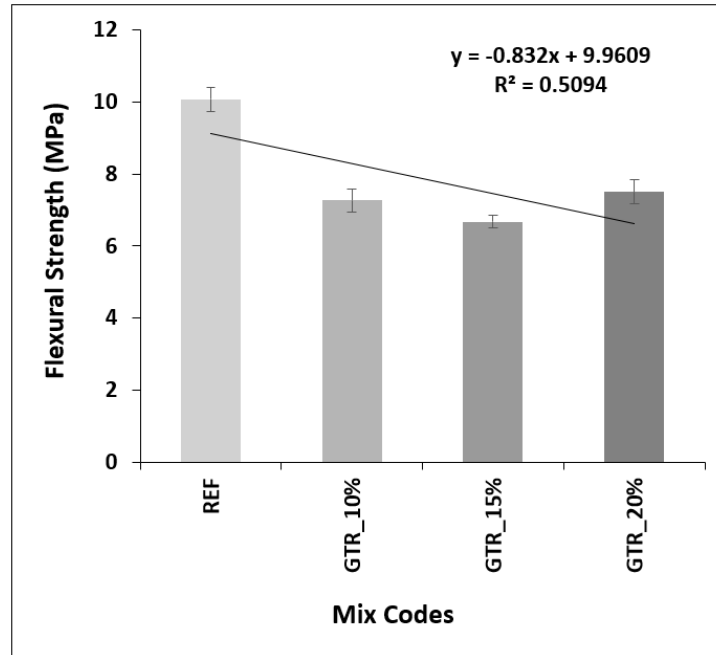
4.6. Flexural strength

Table 13 presents the numerical results obtained in the laboratory for the flexural strength test on the samples. The values obtained represent the influence of rubber and vinyl acetate on the flexibility of the mortars. Figure 46 illustrates the flexural tensile strength of the reference Specimens and those with untreated rubber. It can be observed that the values decrease when rubber is added, reaching a reduction of more than 33.70% in the flexural tensile strength values.

Table 13 – Flexural strength test results at 28 days

MIX Code	Flexural Strength (MPa)	Standard Deviation (MPa)	CoV (%)
REF	10.08	0.33	3.29
GTR_10%	7.27	0.33	4.56
GTR_15%	6.68	0.17	2.48
GTR_20%	7.50	0.33	4.42
KMnO4_10%	8.55	0.83	9.69
KMnO4_15%	9.14	0.99	10.88
KMnO4_20%	8.32	0.83	9.96
VA_REF	4.14	0.49	11.78
VA_GTR_10%	3.87	0.12	3.03
VA_GTR_15%	6.33	0.12	1.85
VA_GTR_20%	6.80	1.33	19.51
VA_KMnO4_10%	5.74	0.17	2.89
VA_KMnO4_15%	6.80	0.33	4.88
VA_KMnO4_20%	6.09	0.47	7.69

Figure 46 – Flexural strength for the reference mixtures and mixtures with untreated GTR addition



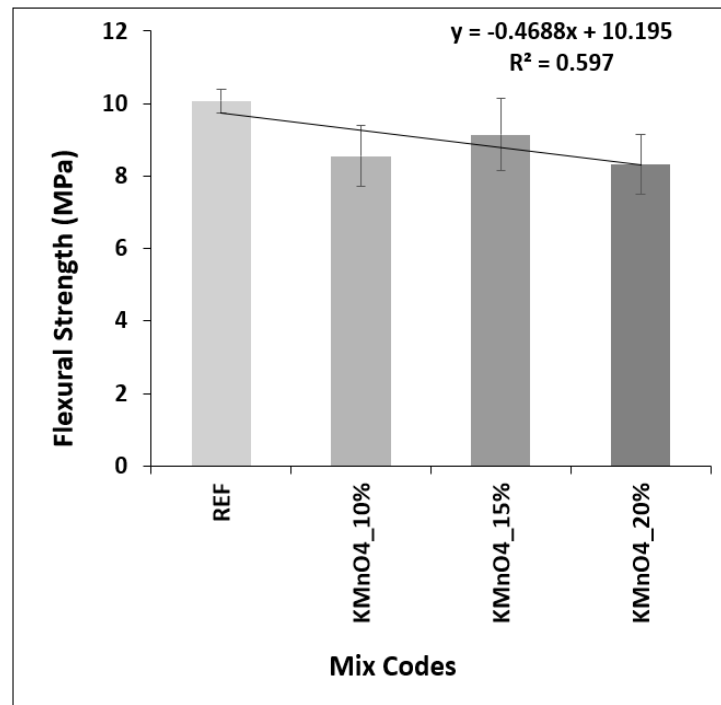
Source: (Author, 2023)

The results of the specimens with untreated rubber demonstrate a similar trend as found in compressive strength. This is due to the same reasons mentioned before, the hydrophobic nature of the rubber that affects the adhesion of the recycled fine aggregate to the cement paste, resulting in a weak interface in the transition zone. This weak interface contributes to a reduction in both compressive and flexural tensile strength values when rubber is added to the cementitious matrix.

On the other hand, the addition of treated rubber reduces the detrimental effects on flexural tensile strength, as presented in Figure 47. The maximum reduction obtained was 17.44% for a 20% volumetric fraction of sand replacement with treated GTR. For a 15% replacement,

The flexural tensile strength was augmented significantly by using treated GTR instead of the untreated rubber, where the augmentation registered 15.07, 26.92 and 9.86% for the 10, 15 and 20% replacements of rubber by sand proportions. The same reasoning in the augmentation of compressive strength applies to the flexure strength, as well. The oxidation potential of potassium permanganate influences the GTR surface with polarity due to introducing polar functional groups on its surface, enhancing the hydrophilicity. Consequently, the water mobility within the mixture is enhanced, which leads to higher extent of cement hydration on the rubber's surface.

Figure 47 – Compressive strength for the reference mixtures and mixtures with KMnO₄ surface treatment.

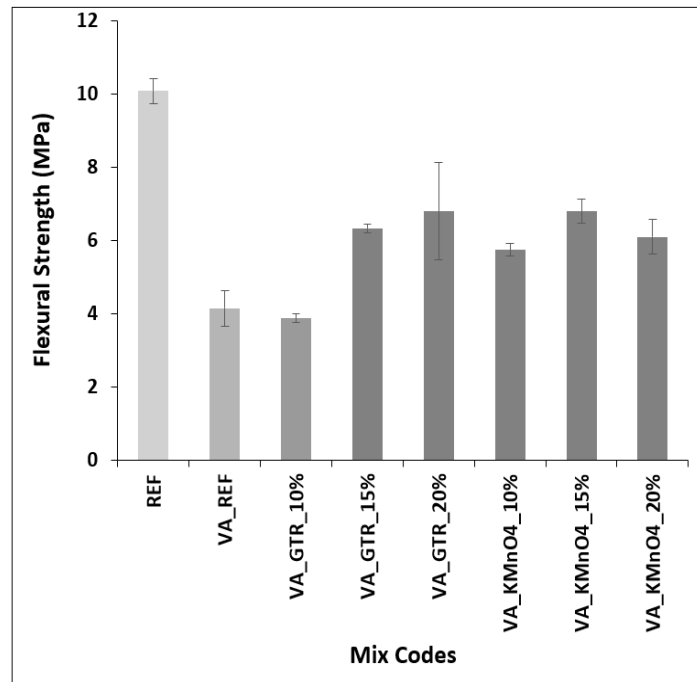


Source: (Author, 2023)

In Figure 48, the results of the flexural tensile strength of the specimens with treated and untreated rubber with the addition of VA are presented. A reduction in the flexural tensile strength was observed, consistent with the results obtained in the specimens with rubber, as well. However, due to the formation of bubbles in the fresh state mixture, which hindered its compaction during molding the specimens, a non-linear response of the values was highlighted. The presence of VA in the mixture contributes to the formation of air voids, leading to a decrease in the flexural tensile strength of the cementitious composites, in accordance with the results of compressive strength of the same mixtures.

Worth mentioning that the flexural strength of the specimens with VA additive demonstrated an increasing tendency when VA was added with treated and untreated GTR, which may indicate that the fine particles of GTR acted as a filler and partially reduced the void content, leading to a slight increase in compressive strength (see Figure 48).

Figure 48 – Flexural strength for the reference mixtures and mixtures with treated and untreated GTR and EVA addition



Source: (Author, 2023)

4.7. Dynamic elastic modulus

Table 14 displays the numerical results of the dynamic modulus of elasticity of the tested specimens. These values provide valuable insights into how rubber influences the specimen's deformability under load conditions.

From the results, it is evident that the modulus of elasticity decreases when untreated rubber is introduced in comparison to the control mix, as depicted in Figure 49. The modulus is shown to be 30,24%, 35,57%, and 38,60% lower than the control specimens when a volumetric replacement of the sand by GTR occurs in 10%, 15%, and 20%, respectively.

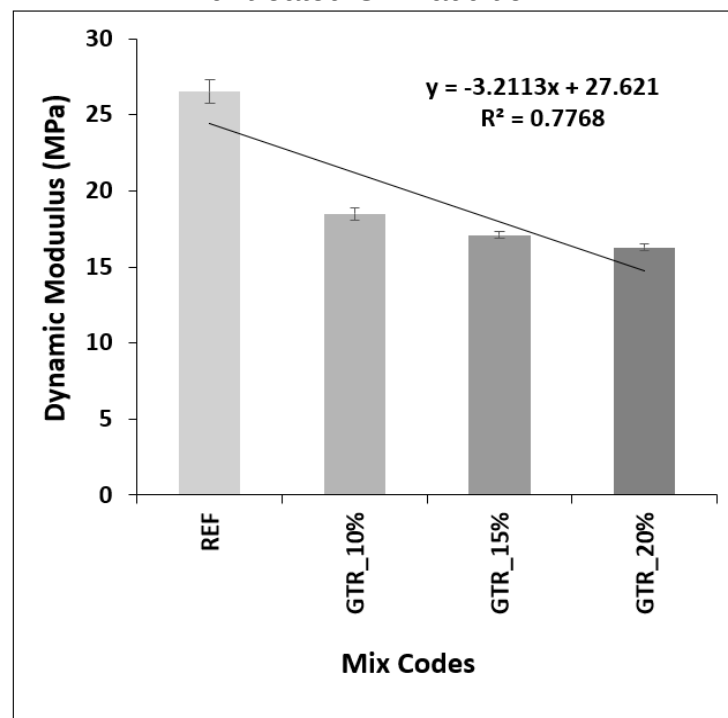
The obtained results are justified by the different deformation capacities of the applied fine aggregates: sand and ground tire rubber. Additionally, the weak interface between the aggregate and cement paste allows greater deformability of the mixture in the hardened state, as the cement hydration is hindered due to the hydrophobic nature of the rubber, thereby reducing the hydration products that contribute to increase the mechanical responsiveness of the specimens under stress limiting their

deformability in the absence of GTR particles, which means maintaining a dynamic elastic modulus value close to the control specimens. This effect highlights the rubberized composites to be applied in environments that require low mechanical properties and high deformability.

Table 14 – Dynamic elastic modulus test results at 28 days

MIX Code	Modulus (MPa)	Standard Deviation (MPa)	CoV (%)
REF	26.51	0.79	2.99
GTR_10%	18.50	0.40	2.19
GTR_15%	17.08	0.22	1.30
GTR_20%	16.28	0.23	1.38
KMnO4_10%	19.79	0.21	1.08
KMnO4_15%	17.94	0.23	1.30
KMnO4_20%	16.48	0.18	1.11
VA_REF	12.42	1.54	12.44
VA_GTR_10%	14.71	1.07	7.29
VA_GTR_15%	14.15	0.65	4.58
VA_GTR_20%	13.65	0.44	3.20
VA_KMnO4_10%	11.85	0.30	2.53
VA_KMnO4_15%	12.91	0.17	1.29
VA_KMnO4_20%	10.38	0.51	4.87

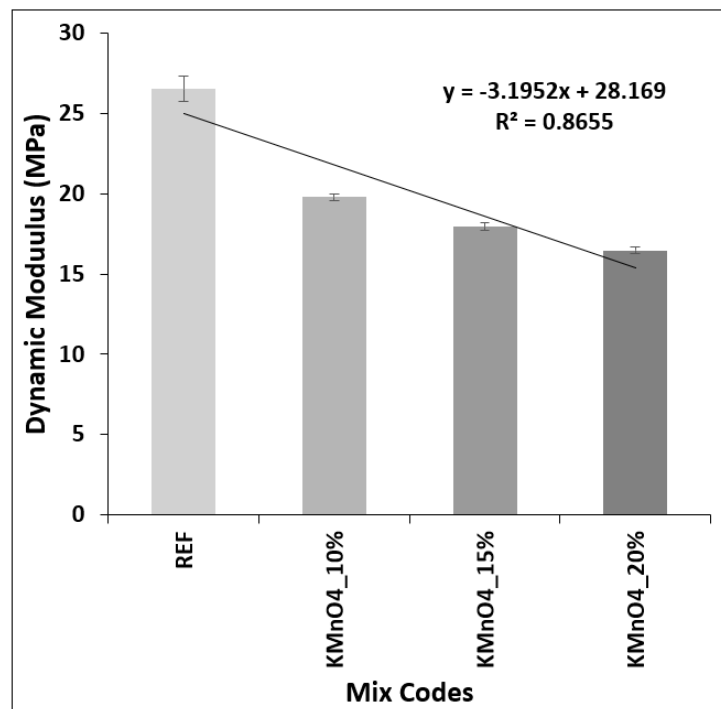
Figure 49 – Dynamic elastic modulus for the reference mixtures and mixtures with untreated GTR addition



Source: (Author, 2023)

Figure 50 displays the results of the dynamic modulus of elasticity test for the reference specimens and those with treated GTR. The obtained values of the specimens with treated GTR were lower than those of the reference sample; however, they are higher than the mixtures with untreated GTR. The dynamic modulus values are 25.36%, 32.33%, and 37.85% lower than the reference sample for sand substitutions of 10%, 15%, and 20%, respectively. On the other hand, it is observed that the values for the same amount of treated rubber, compared to untreated rubber, are higher, ranging from 1.21% to 6.54%.

Figure 50 – Dynamic elastic modulus for the reference mixtures and mixtures with treated and untreated GTR and EVA addition



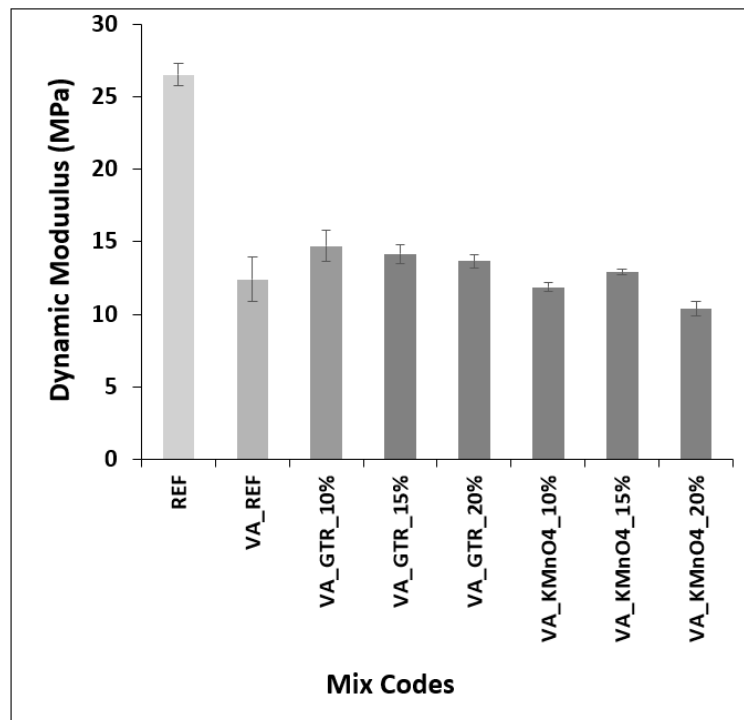
Source: (Author, 2023)

Indeed, the higher values observed in the treated samples compared to the untreated ones can be attributed to the increased hydrophilicity of the rubber, which allows better cement hydration at the interface of the transition zone. The treatment with potassium permanganate enhances the affinity of the rubber for water, improving its interaction with the cementitious matrix during the hydration process. This, in turn, promotes a stronger bond between the rubber and the cement paste, leading to

enhanced mechanical properties and higher dynamic modulus values of the specimens with treated GTR.

Figure 51 shows the values of the dynamic elastic modulus of the specimens with vinyl acetate without GTR, with treated GTR, and with untreated GTR. It can be observed that the values remain lower than the control sample, and there is a non-linearity of the specimen's responsiveness due to the previously reported difficulties during the compacting process caused by the nature of the emulsion additive within the matrix, which generated bubbles in the during the blending process.

Figure 51 – Dynamic elastic modulus of the reference mixtures and mixtures with treated and untreated GTR and EVA addition.



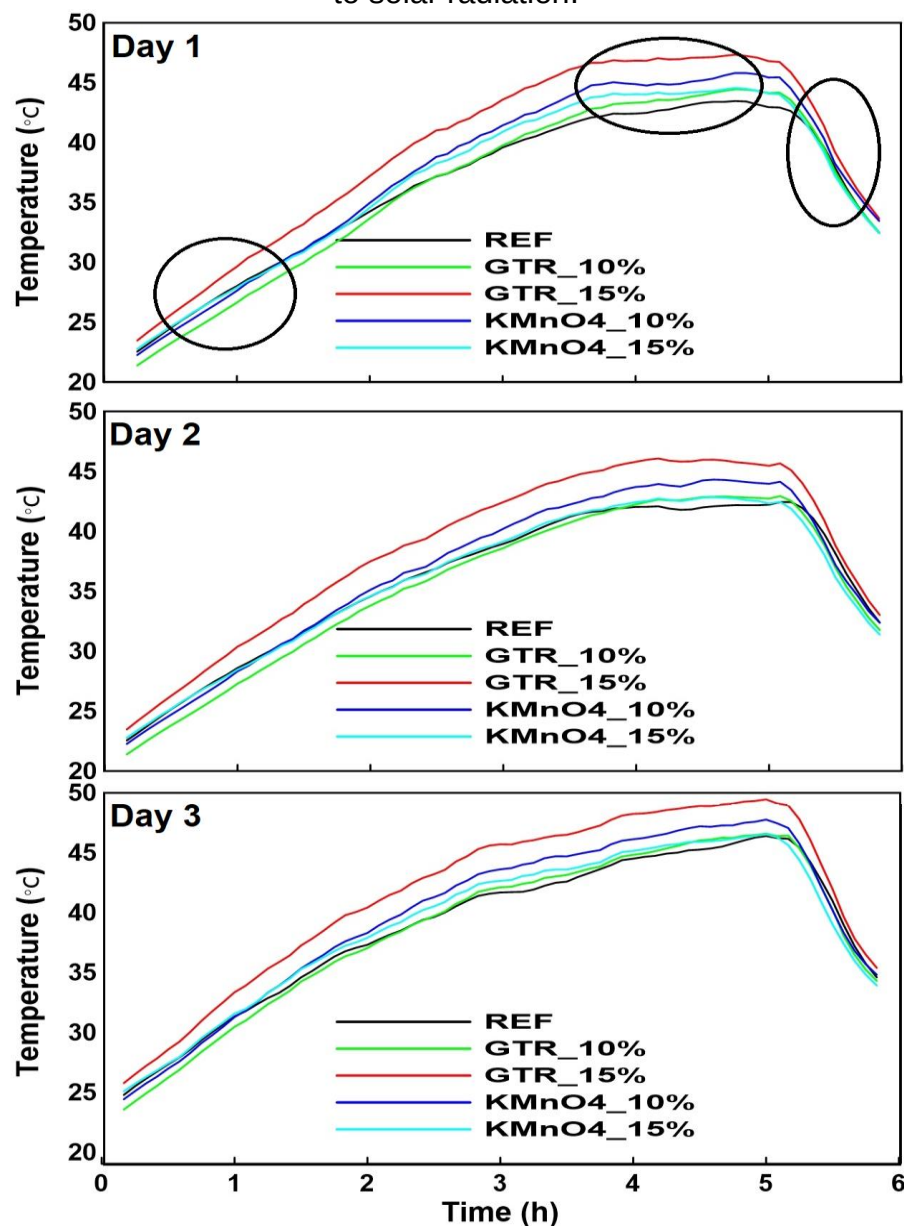
Source: (Author, 2023)

4.8. Thermal Behavior in Solar Radiation

Figure 52 shows the first set of the test during three days monitoring only the exposed side to the solar radiation during day and night. For data optimization, the author explored the thermal response of the thermocouples during 6 hours only from 9 am to 03 pm during 3 days.

The surface temperatures of all types of floor boards increase with time, which is expected due to solar radiation causing heat absorption. There are fluctuations in temperature across all types of floor boards, which could be influenced by factors like changes in solar intensity, ambient temperature, and other environmental conditions. Around the 5-hour mark, the temperatures of all floor boards seem to stabilize and reach an equilibrium state. This might indicate that the thermal dynamics of the materials have reached a balance between heat absorption and heat dissipation.

Figure 52 – First set: Monitoring of the surface temperature of floor boards exposed to solar radiation.

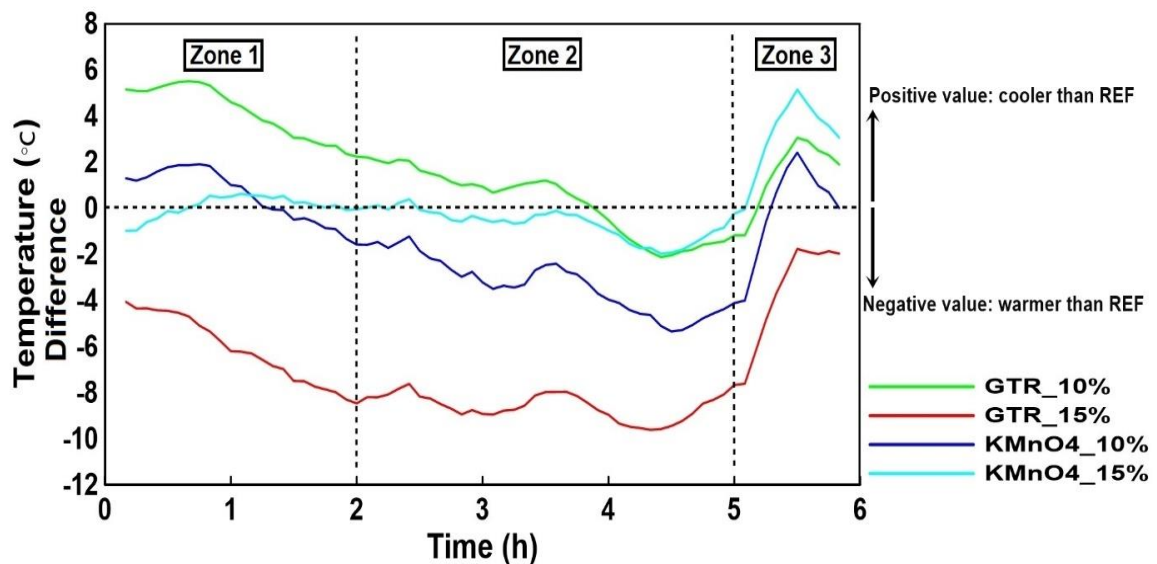


Source: (Author, 2023)

For further analysis and better understanding of the thermal behavior of the rubberized boards, the surface temperature differences between the rubberized boards and the control board REF during 6 hours of exposure to solar radiation were calculated and illustrated in Figure 53.

Figure 53 illustrates a dataset showing the surface temperature differences between the rubberized boards (GTR_10%, GTR_15%, KMnO₄_10%, KMnO₄_15%) and the control board REF during a 6-hour exposure to solar radiation. The values in the dataset represent the temperature differences in degrees Celsius for each treatment compared to the control board (REF) at various time intervals.

Figure 53 – Surface temperature differences between the rubberized boards and the contole board REF during 6 hours of exposure to solar radiation.



Source: (Author, 2023)

At the start (Zone 1: from 9 am to 11 am), the temperature differences are positive for "GTR_10%" and "KMnO₄_10%", indicating that they are slightly cooler than the control board (REF). Conversely, "GTR_15%" and "KMnO₄_15%" have negative temperature differences, implying they are warmer than the control. As time progresses, the temperature differences fluctuate. Both positive and negative temperature differences are observed for all treatments over the 6-hour period.

"GTR_10%" treatment consistently maintains positive temperature differences, indicating that it remains slightly cooler than the control board (REF). "GTR_15%" treatment maintains negative temperature differences, implying it remains warmer than the control. "KMnO₄_10%" treatment starts with a positive temperature difference, but

the differences become smaller and eventually go negative, suggesting it becomes warmer than the control toward the later time intervals. "KMnO₄_15%" treatment has a similar trend as "KMnO₄_10%", with the temperature differences eventually becoming negative (Zone 2: from 11 am to 02 pm).

"GTR_10%" appears to be consistently cooler than the control board throughout the 6-hour period, indicating that this rubberized board effective in reducing temperature rise, though the temperature differences are relatively small. "GTR_15%" is consistently warmer than the control board. "KMnO₄_10%" and "KMnO₄_15%" treatments initially show warming effects, but their temperature differences trend toward becoming cooler than the control board.

At the 5-hour mark (Zone 3: from 02 pm to 03 pm), the rubberized boards (GTR_10%, KMnO₄_10%, KMnO₄_15%) consistently maintain positive temperature differences, indicating that they remain slightly cooler than the control board (REF). This might indicate that the thermal dynamics of the materials have reached a balance between heat absorption and heat dissipation.

The obtained results indicate minimal variation in the surface temperature of the studied boards when rubber is added as fine aggregate. However, the boards with rubber addition showed an average increase in surface temperature of approximately 0.43°C compared to the reference sample, representing less than 2% increment. Statistical analysis of ANOVA indicates that the explored results averages of the surface temperature of all the boards are not significantly different. This means that the discussed results in this section do not present a clear influence of the GTR, treated or not, on the thermal behavior of the studied floor boards, under the applied conditions of the test.

Several factors may not have led to the anticipated thermal insulation or cooling effect of the rubberized boards. Among these factors, the following are noteworthy:

1. Heterogeneity in Board Composition: There's a possibility that the thermocouple was incorrectly positioned on the board, or the ground tire rubber (GTR) wasn't thoroughly mixed within the board material. This inconsistency in composition could affect the thermal properties.
2. Limited Measurement Points: The thermocouple's placement may not cover a sufficient measuring area, potentially failing to represent the entire board's thermal behavior accurately.

3. Fine Particle Size of Applied GTR: The GTR utilized has a particle size of 120 mesh, which might limit its effectiveness as a thermal insulating component. Larger particle sizes are generally more conducive to effective insulation.
4. Limited Sensitivity of Thermocouples: The thermocouples used may have low sensitivity, making them less responsive to subtle temperature changes, thus affecting the accuracy of temperature measurements.
5. Weather Conditions: External weather conditions, such as solar radiation and ambient temperature, can influence the board's thermal behavior. These conditions could have played a role in counteracting the expected cooling effect.

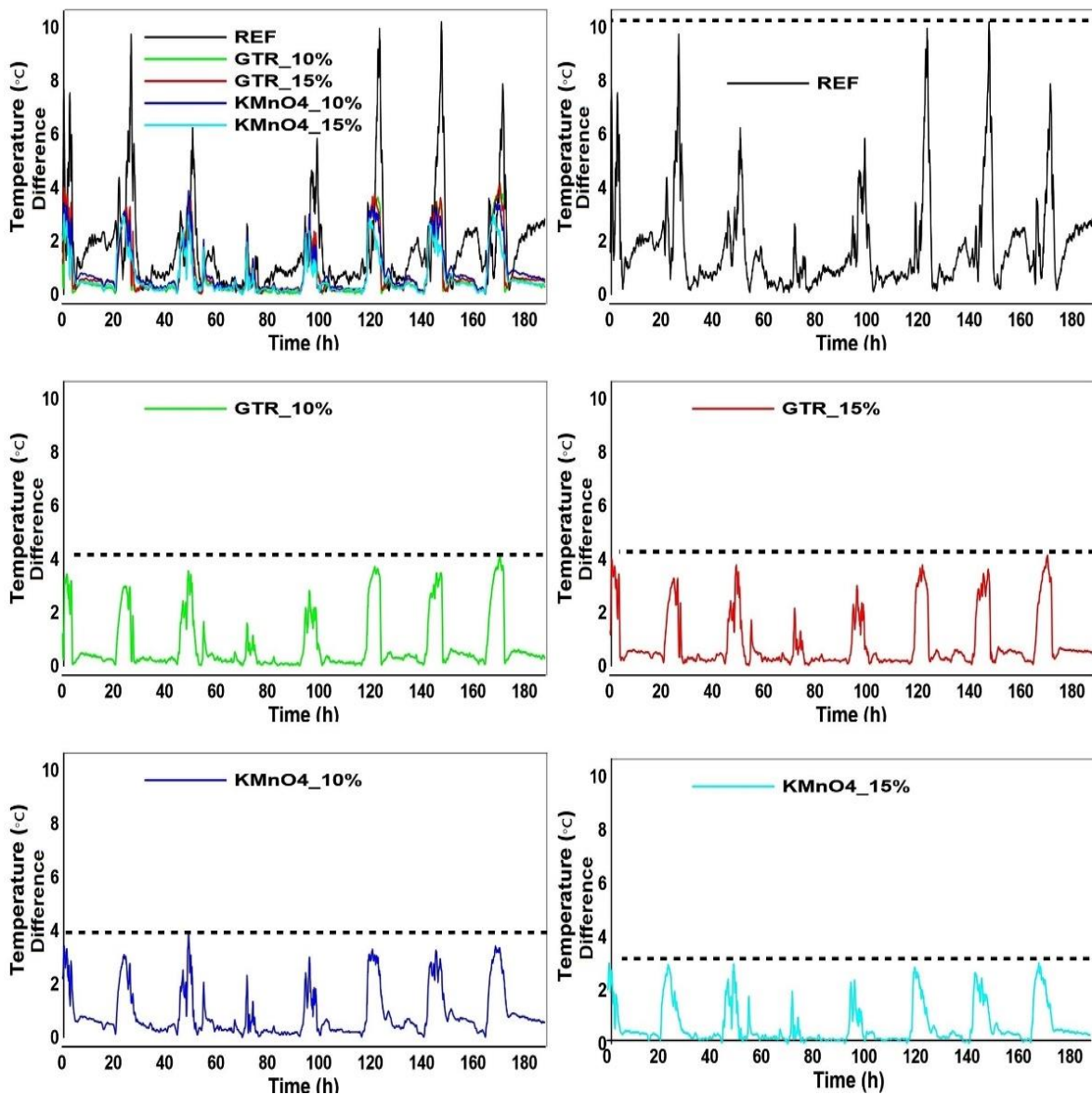
These factors collectively point to potential reasons why the rubberized boards did not exhibit the intended thermal insulation or cooling effects as initially anticipated. Further investigation into these aspects, as well as addressing these considerations, could provide a more accurate understanding of the observed outcomes. depicts the results of the second set of testing conducted over a period of eight days. This phase involved monitoring the heat flux moving from one side of the boards to the opposite side. This heat transfer is represented by the temperature difference observed between the two sides. The objective of this second set of tests was to examine the behavior of the boards under different conditions—both with and without rubber—regarding their ability to facilitate heat transfer from one side to the other, and to consider extending the study duration to see if the trends observed in this 6-hour period persist or change over longer exposure times.

Statistical analysis of ANOVA indicates that the explored results of the heat transfer, represented by the temperature difference observed between the two sides of the boards, are significantly different. This signifies a substantial variation in heat transfer effectiveness across the different experimental conditions. In accordance with thermal analysis interpretation, these findings imply that the factors being investigated have a substantial influence on the heat transfer process.

The observed significant difference in temperature differences between the board's sides goes beyond random fluctuations and indicates that specific variations in insulation types and thicknesses directly affect heat transfer. Consequently, it can be deduced that the experimental treatments directly impact the boards' thermal

conductivity and their ability to regulate temperature differences effectively. This knowledge is vital for engineering applications where managing heat transfer is a critical factor. The aforementioned factors contributing to the fluctuance of the results are also relevant in this test. This fluctuance could be attributed to the inherent characteristic of rubber, which exhibits a lower thermal conductivity value (k) when compared to traditional aggregates used in insulation materials. Rubber, often being a more insulative material, possesses a lower ability to conduct heat compared to conventional aggregates.

Figure 54 – Second set: Monitoring the heat flux transferred from the exposed side. to the other one, represented by the difference of the temperatures of both sides.



Source: (Author, 2023)

5. CONCLUSIONS

The performed tests demonstrated that KMnO_4 improved the hydrophilicity of GTR and had no influence on the crosslinked networks. Additionally, H_2O_2 did not significantly affect the polarity of GTR, as evidenced by FTIR and the contact angle results. Time was found to be a critical variable for treatment efficacy.

Potassium permanganate (KMnO_4) is a powerful oxidizing agent. When it encounters the rubber surface, an oxidation reaction occurs, in which KMnO_4 introduces polar functional groups to the polymeric structure of the rubber. This oxidation leads to the formation of polar functional groups on the surface of the rubber, such as hydroxyl groups ($-\text{OH}$) and carbonyl groups ($\text{C}=\text{O}$). These polar groups make the rubber surface more hydrophilic, meaning it is more ready to interact with water and polar substances.

This improvement in the hydrophilicity of the rubber treated with potassium permanganate can be beneficial in various applications, especially in cementitious mixtures, where interaction with water is essential for better cement hydration and improved mechanical properties.

In addition, the obtained results allowed us to understand the effects of adding treated and untreated rubber, as well as vinyl acetate, in cementitious matrices, leading to the following conclusions:

- I. The addition of treated or untreated rubber did not significantly impact water absorption. However, the addition of vinyl acetate hindered mortar compaction, increasing its permeability due to the formation of bubbles caused by the blending of the emulsion containing surfactant agents.
- II. Samples with rubber are lighter compared to the reference samples since the bulk density of rubber is lower than that of the commonly used fine aggregate. However, the use of pre-treated rubber increased the specific weight of the mortars as it enhanced rubber hydrophilicity, optimizing water migration in the mixture, and thereby improving cement hydration and the production of hydration products.
- III. There is a significant reduction in compressive strength for samples with untreated rubber, with improvements observed when using pre-treated rubber. This is attributed to the change in the elastomer's polarity, which

increases water affinity and enhances cement paste hydration. Nevertheless, the Brazilian standard ABNT NBR 6.118/2014 establishes a minimum compressive strength of 20 MPa for concrete structures, a value surpassed by all samples except those with vinyl acetate. Therefore, the use of rubber in flooring slabs or raised floors appears to be structurally viable.

- IV. Flexural tensile strength decreased with the addition of untreated rubber, while the use of treated rubber resulted in values close to the control sample.
- V. Dynamic modulus of elasticity results supports previous research findings, recommending the application of rubberized mortars and concretes in scenarios that require impact resistance due to the decrease in this property, providing greater deformability to the samples.
- VI. The use of rubber in cementitious matrices to reduce the surface temperature of mortars exposed to solar radiation is not viable. Despite rubber being an excellent thermal insulator, as indicated in the literature review, it is not effective in reducing surface temperature. Temperature remains a preeminent variable in controlling this property, and recycled rubber from tires has an extremely dark coloration.

Several factors may not have led to the anticipated thermal insulation or cooling effect of the rubberized boards, as mentioned in the discussion section. These factors collectively point to potential reasons why the rubberized boards did not exhibit the intended thermal insulation or cooling effects as initially anticipated. Further investigation into these aspects, as well as addressing these considerations, could provide a more accurate understanding of the observed outcomes.

Indeed, the use of rubber in cementitious matrices proves to be viable for both structural and non-structural applications. Incorporating this recycled aggregate into floor boards can ensure the required mechanical performance and provide enhanced impact absorption due to the material's greater deformability, reflected into the low values of the dynamic modulus of elasticity.

Moreover, employing rubber for this purpose would help mitigate the adverse effects of improper tire disposal, which negatively impacts sustainable development, harming not only the environment but also public health. By reusing rubber in cementitious applications, we can contribute to more sustainable practices, reducing waste and promoting environmental responsibility.

Overall, the use of rubber in cementitious mixtures presents a promising solution, addressing both technical and environmental challenges, and promoting a more sustainable construction industry.

6. FUTURE WORK SUGGESTIONS

Conducting a comprehensive study of the thermal behavior of rubberized floor boards using advanced techniques and various parameters would provide valuable insights into their performance. Here's a some of the proposed future work:

- Ultra-Sensitive Sensors and Thermal Behavior:

Implementing ultra-sensitive sensors that cover a substantial area of the specimen would enable high-resolution temperature monitoring. This advanced sensor setup could help capture detailed temperature profiles across the rubberized floor boards during heat transfer experiments. By obtaining more precise data, researchers can better understand the intricate heat distribution patterns and potential variations across the material.

- Effect of Particle Size Variation:

Introducing rubberized specimens with different particle sizes of the Ground Tire Rubber (GTR) is a crucial step. This parameter can significantly impact the material's thermal properties. Conducting experiments with varying particle sizes would shed light on how GTR particle size influences heat transfer within the rubberized floor boards. This information could guide the formulation of optimized rubberized materials for specific applications.

- Controlled Weather Conditions:

Using a closed chamber to control weather conditions is an excellent approach. Maintaining consistent and controlled environmental conditions during experiments helps eliminate external factors that could affect heat transfer results. This controlled setup ensures that variations in temperature, humidity, and airflow do not confound the interpretation of experimental data.

- Impact Resistance Testing:

Evaluating the impact resistance of rubberized specimens is essential to understand the material's durability and potential applications. By subjecting the treated rubber to impact tests, researchers can assess its resilience and behavior under dynamic

loading conditions. This information is valuable for applications where the material might experience impacts or mechanical stress.

➤ Deformation Analysis with LVDTs:

Using Linear Variable Differential Transformers (LVDTs) to monitor the deformation of rubberized specimens provides insights into the material's mechanical response under different conditions. This data can be used to assess the material's potential for various applications that involve deformation, such as vibration isolation, structural damping, or flexible components.

➤ Multidisciplinary Insights:

The proposed study combines aspects of material science, thermal analysis, mechanical testing, and instrumentation. The multidisciplinary approach allows researchers to gather a comprehensive understanding of the rubberized floor board's behavior, enabling well-informed decisions for future applications and material improvements.

Incorporating these advanced techniques and comprehensive analyses will contribute significantly to the knowledge of rubberized materials' thermal and mechanical properties, leading to their more effective and optimized use across various domains.

7. RELEVANT PRODUCTIONS

7.1. Conference abstract

RODRIGUES ANDRÉ, F.; GALAL ABOELKHEIR, M. **Sustainable approach of applying previous treatment of tire wastes as raw material in cement composites: Review**. Materials Today: Proceedings. Anais... Em: 3RD INTERNATIONAL CONGRESS ON MATERIALS & STRUCTURAL STABILITY. Rabat: 2 abr. 2022. Acesso em: 20 abr. 2022

ANDRE, F. R.; GARCIA, A. I. Z. ; SILVA, D. P. ; SILVA, G. A. ; MARCOLONGO, G. ; RAMBO, D. S. ; ABOELKHEIR, M. G. A. M. . O avanço do emprego de borracha de pneu como agregado reciclado na construção civil. In: 1ª Conferência Brasileira de Planejamento Experimental e Análise de Dados (ConBraPA-2020-Virtual), 2020, Virtual. Anais da 1ª Conferência Brasileira de Planejamento Experimental e Análise de Dados (ConBraPA-2020-Virtual), 2020.

7.2. Journal Paper

RODRIGUES ANDRÉ, F.; GALAL ABOELKHEIR, M. Sustainable approach of applying previous treatment of tire wastes as raw material in cement composites: Review. Materials Today: Proceedings, 2 abr. 2022.

8. APPLICATION MANUAL

8.1. Rubber treatment

The treatment of rubber involves the separation of a quantity of material ground to a mesh size of 120, immersion in an aqueous solution of sodium hydroxide with a concentration of 10% for 40 minutes, and filtration to separate the solution from the rubber. After the filtration process is complete, the material should be washed, and the pH should be checked until it reaches neutrality. Once this step is completed, the rubber should be dried in an oven until it reaches zero moisture content.

The material washed in the sodium hydroxide solution should undergo an immersion treatment with potassium permanganate added in an aqueous solution at a concentration of 5% and maintained at 60°C for 3 hours. At the end of the immersion period, the material can be filtered and placed in an oven for complete drying.

8.2. Preparation of floor board

The rubberized mortar floor board will be prepared using Portland cement type CP-V, natural sand, treated mesh 120 rubber, water, and a superplasticizing additive. The mixture ratio to be adopted should follow the following proportion 1:3:0.52:0.007 (cement: sand: water: superplasticizing additive), with the volumetric fraction of sand replaced by GTR amounting to 10%.

The process of mixing materials and compaction should follow the methodology presented in the ABNT NBR 16.738 standard. Curing should take place in a moist chamber for 28 days with controlled temperature.

The thickness and dimensions of the boards can vary depending on the needs of each application and can be dimensioned using the properties of the composite.

8.3. Physical-mechanical properties of the floor boards

The floor boards exhibit characteristics as described below, which should be taken into account in their design to ensure the safety of their application:

- Compressive strength (28 days): 39.73 MPa;
- Flexural strength (28 days): 8.55 MPa;
- Dynamic modulus of elasticity (28 days): 19.79 MPa;
- Water absorption (28 days): 8.74%;
- Real specific mass (28 days): 2.21 g/cm³.

8.4. Application of the floor boards

The floor boards can be applied either laid on a cement and sand mortar or on a raised floor structure, considering the specific requirements of each construction project.

For the application of the floor on a cement and sand mortar, the following standard recommendations should be followed:

- a) Prepare a leveled surface on the subfloor for the laying of the floor;
- b) Saturate the substrate with water for better adhesion;
- c) Apply dry cement and sand mortar on the damp substrate;
- d) Gradually add water to the dry cement and sand mortar to moisten it;
- e) Lay the floor on the base created using a rubber mallet and leveling laths;
- f) Maintain a spacing for grouting between 4 and 5mm;
- g) Perform grouting evenly with the help of a grout trowel without interruptions, in a single pass;
- h) Prevent moisture from coming into contact with the floor and grout during its curing;
- i) After installation, polishing of the floor can be carried out, followed by waterproofing with a water-repellent material.

For the application of the floor on a raised floor structure, the manufacturer's recommendations should be followed, and it is permissible to use reinforcement adhered to the back of the boards to resist bending tensile forces. The design of the reinforcement should be carried out based on the specific requirements of the application.

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