

**INVESTIGATION OF THE GRINDING PARAMETERS
ON THE POZZOLANIC PROPERTIES OF
CONSTRUCTION AND DEMOLITION WASTE**

**ÖĞÜTME PARAMETRELERİNİN İNŞAAT VE YIKINTI
ATIKLARININ PUZOLANİK ÖZELLİKLERİNİN
ÜZERİNE ETKİSİNİN ARAŞTIRILMASI**

ERAY TEKSİN

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ABSTRACT

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The accumulation of construction debris waste (CWD) and disaster wastes in the environment causes environmental degradation and natural resources are consumed for the reconstruction of demolished areas. Construction demolition wastes contain different types of materials due to their inherent characteristics. These types of materials consist of hazardous and recyclable elements. While various construction materials such as aggregate, concrete, bricks, wood, iron can be recycled by separation facilities, hazardous chemicals such as asbestos and phenol need to be intervened appropriately.

Recycled materials can be reused as raw materials in different sectors, contributing to the circular economy. Proper recycling of waste materials resulting from demolition and disasters can address the raw material needs to produce concrete and cement, fundamental building blocks in the construction sector. Although the materials generated as a result of disasters and demolitions can be recycled, the physical and chemical properties of the waste vary depending on the location. Therefore, comprehensive studies are essential for evaluating and discovering the potential of these waste materials.

The subject of this thesis is to observe the pozzolanic activity of brick waste, a type of construction demolition wastes, by the effect of different grinding parameters. The influence of different material filling ratios and grinding durations on the pozzolanic activity of brick waste is studied in this thesis. Mechanical and chemical tests are carried out to observe the pozzolanic activity. It is aimed to improve the pozzolanic activation of the waste material by optimizing the grinding parameters. Thus, it is believed that the use of brick waste as a substitute for cement will pave the way for the use of waste in the cement industry and provide a more sustainable and eco-friendly solution.

Keywords: Grinding Parameters, Construction and Demolition Waste, Brick Waste, Pozzolanic Activity, Material Filling Ratio, Ball Filling Ratio, Grinding Duration

ÖZET

ÖĞÜTME PARAMETRELERİNİN İNŞAAT VE YIKINTI ATIKLARININ PUZOLANİK ÖZELLİKLERİNİN ÜZERİNE ETKİSİNİN ARAŞTIRILMASI

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İnşaat yıkıntı atıklarının (İYA) ve afet atıklarının çevreye yığılması neticesinde çevreye zarar verilmekte, yıkılan alanların inşası için doğal kaynaklar kullanılmaktadır. İnşaat yıkıntı atıkları özellikleri gereği bünyesinde farklı malzemeler bulundurmaktadır. Bunlar, tehlikeli ve geri dönüştürülebilir malzemelerden oluşmaktadır. Agregası, beton, tuğla, ahşap, demir gibi çeşitli inşaat malzemeleri ayrıştırma tesisleri tarafından geri dönüştürülebilirken, asbest, fenol gibi zararlı kimyasallara uygun şekilde müdahale edilmesi gerekmektedir.

Geri dönüştürülen malzemeler farklı sektörlerde hammadde olarak tekrar kullanılabilir ve döngüsel ekonomiye katkıda bulunmaktadır. Yıkıntı ve afet neticesinde ortaya çıkan atıl malzemelerin uygun koşullarda geri dönüştürülmesiyle beraber, inşaat sektörünün temel yapı taşı olan beton ve çimento üretimi için gerekli hammadde ihtiyacı karşılanabilmektedir. Her ne kadar, afet ve yıkıntı sonucunda ortaya çıkan malzemeler geri dönüşüm ile değerlendirilebilse de ortaya çıkan atığın fiziksel ve kimyasal özellikleri buldukları lokasyona göre değişmektedir. Bu sebeple

ortaya çıkan atıkların değerlendirilmesi hususunda kapsamlı çalışmaların yapılması ve potansiyellerinin keşfedilmesi dikkat çekmektedir.

Bu tezin konusu inşaat yıkıntı atıklarından biri olan tuğla atıklarının farklı öğütme parametrelerinin etkisi ile puzolanik aktivesinin gözlemlenmesidir. Tez kapsamında farklı malzeme doluluk oranlarının ve öğütme sürelerinin tuğla atığının puzolanik aktivitesine olan etkisi üzerine çalışılmıştır. Puzolanik aktivitenin gözlemlenmesi için mekanik ve kimyasal testler yapılmıştır. Öğütme parametrelerinin optimizasyonu ile atık malzemenin puzolanik aktivasyonunun iyileştirilmesi hedeflenmektedir. Böylelikle tuğla atığının çimentoya ikame olarak kullanılmasıyla beraber çimento sektöründe atıkların kullanılmasının önünün açılacağına beraberinde daha sürdürülebilir ve çevreci bir çözüm sunacağına inanılmaktadır.

Anahtar Kelimeler: Öğütme Parametreleri, İnşaat ve Yıkıntı Atıkları, Tuğla Atıkları, Puzolanik Aktivite, Malzeme Doluluk Oranı, Bilya Doluluk Oranı, Öğütme Süresi

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1. INTRODUCTION

1.1 General

Due to the benefits that they bring during building construction, both concrete and cement are among the favored materials for establishing a good quality of life at the center of modern society. Concrete, the world's second most utilized product after water resource [1]. In addition, concrete is produced in quantities exceeding ten billion tons every year. Due to its structure concrete has great durability, impermeability, and long service of life. Thus, concrete has long been the topic of investigation and development by chemists, material scientists, and engineers.

Concrete is an essential need of the modern construction industry and is commonly employed in it. Cement output has expanded throughout time with the rising demand for concrete in the industry. As a result of cement manufacture, environmental issues have become more prevalent. On a global basis, the production of cement contributes to 8% of the overall carbon dioxide (CO₂) emissions [2,3]. The thermal decomposition of calcium carbonate during the synthesis of the reactive calcium silicate and aluminate phases which compose the basic structure of cement is one of the primary causes of CO₂ emissions from Portland cement (PC) production. One ton of PC manufacture results in a total emission footprint of approximately 0.8 tons of CO₂ equivalent. The reported amount comprises emissions from the combustion of fossil fuels during the producing cement as well as emissions from the transformation of calcium carbonate to its oxide form [4].

There are various research studies which outline the environmental consequences of processes for cement production [5–8]. According to the studies, the calcination process in cyclones performs an important role in the environmental impact of cement production processes [9,10]. Processes such as raw material preparation and post calcination grinding represent less than 20% of the impact associated with climate change. CO₂ emissions during heating responsible for 60%, directly resulting from the chemical decarbonization of limestone, while 40% of these emissions come from the fuel combustion process [11].

The process of producing cement requires a significant amount of energy. According to the International Energy Agency's (IEA) report titled "Low Carbon Transition in the Cement Industry", the cement sector ranks as the third-largest consumer of industrial energy globally, with an energy consumption of 10.7 EJ (Exajoule), accounting for 7% of total energy use among major industries in the world [12]. The calcination of calcium carbonate, or limestone, accounts for two-thirds of the CO₂ emissions within the cement production process, while the remaining one-third is the fuel used for the process. Although there have been significant improvements in energy efficiency, alternative fuel use and clinker substitution within the industry, the cement industry was the second largest global industry in terms of CO₂ emissions in 2014, with annual CO₂ emissions of 2.2 Gt (Gigatonnes), reaching 27% of total industrial CO₂ emissions. Globally, the demand for cement and concrete is increasing with growing populations, changes in urbanization trends and growing infrastructure needs. Hence, current cement production is expected to increase up to 23% by 2050.

In addition to the CO₂ emissions from the cement production process, sulfur dioxide, nitrogen oxides and carbon monoxide are emitted into the atmosphere, causing damage to health and the environment. Nitrous oxide (NO_x) causes environmental impacts such as ground level ozone formation, acid rain, global warming, decreased water quality and causes various health problems such as visual impairment, while high amounts of sulfur dioxide (SO₂) negatively affect the respiratory system and cause an increase in various respiratory and cardiovascular diseases. In addition, SO₂ contributes to the formation of acid rain. Carbon monoxide (CO) is similarly harmful to the environment and human health as NO_x and SO₂ gases. CO gas reduces the amount of oxygen carried to the organs and damages the central nervous system. Furthermore, CO increases the formation of ground-level ozone, causing problems in the respiratory tract [13]. Considering the impact of harmful gases emitted during cement production, increased cement production can cause serious damage to people's quality of life and the environment.

The global world is focusing on concrete production to fulfill the infrastructure requirements of the developing world [14]. Considering that the primary component of concrete is cement, increasing concrete production will raise the demand of the cement.

Despite the fact that concrete is inexpensive, easily accessible, and highly effective, the need for alternative building materials to cement becomes more apparent when its effect on the environment and human health is considered. Researchers worldwide are actively working on developing and implementing alternative binder systems to mitigate the environmental impact associated with cement production. Considering the increasing demand for cement in the world, the production of alternative binders to cement has become crucial in recent years [15]. For this reason, it is necessary to design and produce sustainable and more environmentally friendly building materials to meet the increasing need for cement and concrete in the developing world.

1.2 Objectives and Scope

Main objectives to achieve of this thesis are specified below:

- Investigation of the effects of grinding parameters on pozzolanic properties of CDW based brick waste materials.
- Defining efficient material filling ratio (MFR) and grinding duration at constant ball filling ratio (BFR) as grinding parameters to achieve pozzolanic property on CDW based brick waste.
- Evaluating criteria of activation of the waste material under influence of grinding process by various test methodologies.
- Investigating strength and consistency effects of the activated waste material on mortar which is substituted in the cementitious system.

The scope of this thesis includes effect of different grinding parameters such as MFR and BFR. The filled area, in other words BFR, which represents approximately 20% of the total area, indicates the volume of the balls and the spaces between the balls. The volume of the balls was subtracted from the filled area to calculate the volume between the balls. The filling ratio of the remaining empty volume is defined as MFR. Different MFR values are defined as 60, 80, 100 and 120% while grinding duration is defined as 30, 60, 120, 180 minutes at constant BFR which is 20%. In addition, constant ball types and amounts

are utilized during different grinding processes. Five different steel ball sizes are selected including 38.10, 31.75, 25.40, 19.05, 15.87 mm ball diameters for grinding process. A total of 43 balls of 38.1 mm diameter, 67 balls of 31.75 mm diameter, 10 balls of 25.4 mm diameter, 71 balls of 19.05 mm diameter and 94 balls of 15.87 mm diameter are selected. Firstly, MFR effect on pozzolanic property of material is observed and optimum MFR value is determined by considering material's pozzolanic behavior. Secondly, effect of grinding duration on pozzolanic behavior of material at optimum MFR value is observed.

Particle size distribution analysis is carried out for each grinded waste material. Various test methods are conducted to investigate pozzolanic activity of brick waste which are grinded at different parameters. Test methods are divided into two parts: direct and indirect. Compressive strength test of cement-based and waste-based mortar mixes is conducted as indirect method. In addition, effect of grinding parameters on consistency of cement-based mortar mix is also observed. X-Ray diffraction (XRD) analysis of waste-based paste, thermogravimetric analysis (TGA) of waste-based paste, isothermal calorimetry analysis and Frattini test are carried out as direct method. Grinded waste materials at different parameters are compared for pozzolanic activity. Thus, effective grinding parameters to enhance the pozzolanic properties of the material are observed.

2. LITERATURE RESEARCH

2.1 Investigation of Pozzolanic Reaction

Pozzolanic materials consists of SiO_2 and Al_2O_3 that have little or no cementitious properties by themselves. However, pozzolanic materials can react with calcium hydroxide (CH) at 20 to 25°C and develop hydraulic products. Humanity has been using lime-natural pozzolan mortars for a very long time. Analysis of a concrete slab found in the southern Galilee reveals that the origin of lime and lime pozzolan concrete can be traced back to the Neolithic period, around 7000 BC, rather than the Greek and Roman eras. Throughout the Roman period, this kind of materials were extensively utilized in the construction of aqueducts, retaining walls, building walls and arch bridges. Many Roman monuments, such as the bridges of Fabricus and many maritime monuments, such as and Trajan's arches, credit the durability of lime pozzolan mortars. Mixture of Lime Surkhi which is pulverized fire clay or brick soil has been in common use since Greek and Roman era and are still in use extensively in India. England did not start using lime Dutch truss mixtures until the 17th century. They were extensively used in the Netherlands for the development of harbors and sea defenses. In the construction of numerous buildings in Iceland throughout history, lime and volcanic ash mortars were commonly used. Furthermore, such mortars have lasted for 90 to 400 years due to their high strength and durability [16].

The word of pozzolana has two different meaning. The first meaning includes the pyroclastic rocks which are glassy and zeolitized. The second type of meaning covers inorganic materials which are natural or artificial. Those materials get harden in the composition of water and CH. The utilization of pozzolanas was primarily limited to Italy, which has significant quantities of natural pozzolanas for long period of time. For other countries, requirement for the disposal of artificial pozzolanas such as silica fume and fly ash, has triggered the interest to utilize these materials in the sector [17]. Pozzolanic materials which can be natural or artificial, are used as supplementary cementitious material (SCM). Thus, construction sector consumes less ordinary Portland cement. Today, pozzolanic materials which are used as additives to ordinary Portland cement, consist of solid wastes and industrial byproducts. The reason of utilizing such wastes and byproducts is due to ability of react with lime which occurs during hydration of cement.

There is a fact that pozzolanic reaction products enhance the performance of cement composites [18].

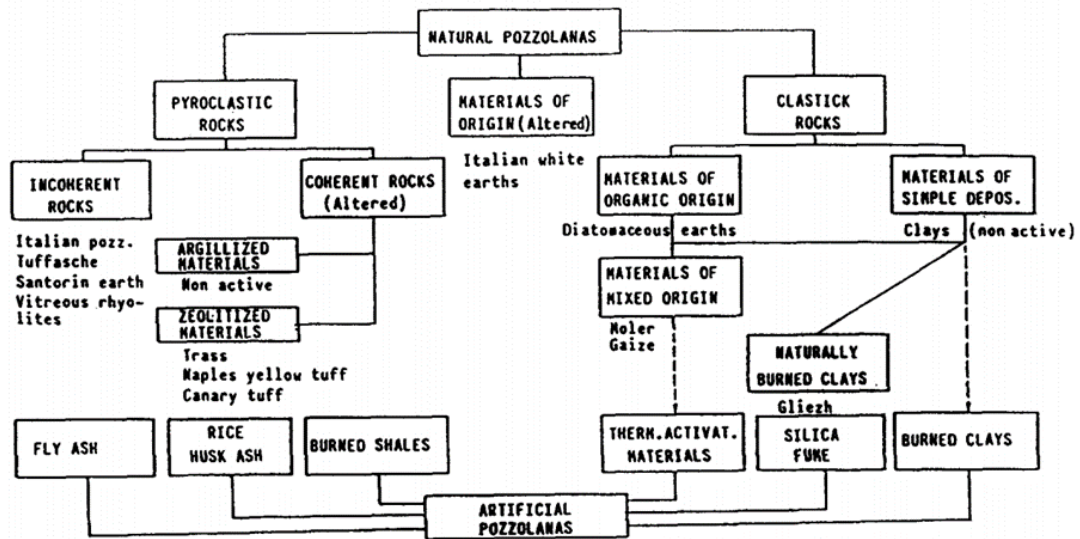


Figure 2.1 Types of pozzolan [17]

In a study on the parameters affecting the pozzolanic reaction of natural zeolites, different types of pozzolans were mixed with water and CH and tested up to 180 days. The materials were grinded and the short- and long-term reactivity of the grinded materials were observed. As a result of the studies, the higher surface area positively affected the short-term reactivity. In addition, it was observed that the long-term reactivity was determined by the Si/Al ratio. It was stated that natural pozzolans with high silica content reacted faster [19].

The pozzolanic activity can be defined as the capacity of silicate or aluminosilicate containing materials to react with CH and water to form reaction products. Pozzolanic activity can be measured by chemical, physicochemical, and mechanical methods. One study investigated the pozzolanic reactivity of brick dust by mechanical and microstructural methods. The compressive strength of mixtures containing brick dust was used as an indicator to measure pozzolanic activity [20]. A study claims that pozzolanic activity of the material is depended on the glassy phase content. The pozzolanic activity is enhanced with an increase in fine granularity and specific surface area. The modified Chapelle test, which is a chemical method based on the amount of CH fixed by the

pozzolan, was used to determine pozzolanic activity. A relationship was established between the compressive strength test and the Chapelle test [21]. The heat of hydration of mortar or concrete can be reduced by pozzolanic admixtures. The effect of the pozzolanic activity of metakaolin on the heat generated during hydration was examined, and its behavior was compared with that of other conventional pozzolanic materials like fly ash and silica fume. The results showed that metakaolin mortars exhibited a slight increase in the heat of hydration compared to mortar which contains only Portland cement due to its higher pozzolanic activity. In terms of heat of hydration, the behavior of metakaolin blended mortar was more like silica fume rather than fly ash [22].

Different test methods were used to analyze the pozzolanic activity. Method of the strength activity index is based on the strength ratio of pozzolanic material and cement mixture on different ages (1,3,7,28). Although it shows the performance of the composite system, this method is time consuming and does not fully reveal the difference between filler effect and pozzolanic reaction. Electrical conductivity test is performed with a mixture of pozzolan and CH. This method can be performed in 2 minutes. Isothermal calorimetry test is performed with pozzolan and cement mixture. Its advantages include rapid, accuracy and high reproducibility, but the equipment cost is high. The thermal analysis method is also fast and can determine activity, but carbonization can easily affect the accuracy of the test. The bound water ratio method is based on the weight loss of pozzolana-cement or pozzolana-lime systems between 105 and 1000 °C. Although it is a fast method, the high temperature can affect the physical and chemical composition of the material. The Frattini test is performed for pozzolan-cement systems. It should be performed with cements with high calcium content. The Chapelle test is performed with pozzolan-lime systems. It is a fast test method, but carbonation affects the process [23]. To conclude, different test techniques were investigated in the literature for the determination of pozzolanic activity under various conditions.

2.2 Mechanochemical Activation on Materials

The paper "On Stones" written in 315 BC by Aristotle's student and successor is recognized as the first document to propose mechano-chemistry [24]. It was discovered that elemental mercury could be acquired by solid-state reaction from cinnabar and acetic

acid by grinding in a mortar and pestle. There is no definitive record of mechano-chemistry for the following 2000 years. Faraday issued literature research on the "dry method" of reducing silver chloride in the presence of zinc, tin, and other metals in 1820 [25]. This work ignited interest in mechano-chemistry. For his methodical investigation on the chemical effects of mechanical action, M. Carey Lea (1823-1897) is considered as the "Father of Mechano-chemistry" [26]. He discovered that mechanochemical processes may yield outcomes that are distinct than standard thermal heating. In 1891, Wilhelm Ostwald described "mechano-chemistry" as one of the four branches of chemistry, alongside photochemistry, thermochemistry, and electrochemistry. [27]. Gerard Heinicke identified mechanochemistry as "a branch of chemistry concerned with the chemical and physical changes that occur in solids under the influence of mechanical action" in 1894. The last two decades have seen the most intense breakthroughs in mechano-chemistry.[28,29].

The most frequently utilized and well-known mechano-chemical techniques involve the processing of various kinds of mills and other similar instruments [30,31]. The outcome is in materials with enhanced surface energy, which causes them to be more reactive. For mechanochemical activation, various types of grinding equipment are utilized. Automatic ball mills, vibrating mills and planetary ball mills, provide more precise control of mechanochemical reactions [27]. In vibrating mills, the reaction chamber oscillates back and forth, leading the enclosed ball bearings to shear and grind the substrates in proximity. In planetary mills, the reaction chamber undergoes high-speed rotation in the opposite direction to the primary rotating "sun wheel," leading to the grinding [32]. The grinding media, grinding speed, ball to power weight ratio, grinding type, grinding area, operating time, and surface contour of the material all influence the efficiency of grinding in the energy-intensive ball milling process. High energy mills perform on distinct principles. Grinding processes are performed through shearing, impacting and compression [33].

The mills and devices used for significant grinding are designed to deliver as much energy as possible to the material which is being ground. This intense energy transmitted to the material leads to degradation on a material-specific basis and thus significantly impacts the reactivity. Taking this into account, it is essential that the mills that will be used for the grinding process should transfer high energy to the material. Abrasive mills, for

example, have a higher energy density than other kinds of grinding equipment. A central shaft equipped with levers consistently blends particles and spherical medium, creating an environment to alter the grinding energy. Additionally, it is crucial that the mill is not only easy to install and use, but also easy to perform maintenance procedures such as cleaning. Ball milling is one of the most valuable instruments for practical mechano-chemical investigations. Extended milling results in enduring plastic deformation due to shear stress. This process creates new surfaces of reactants featuring active zones, facilitating easier contact, combination, and reaction [34,35].

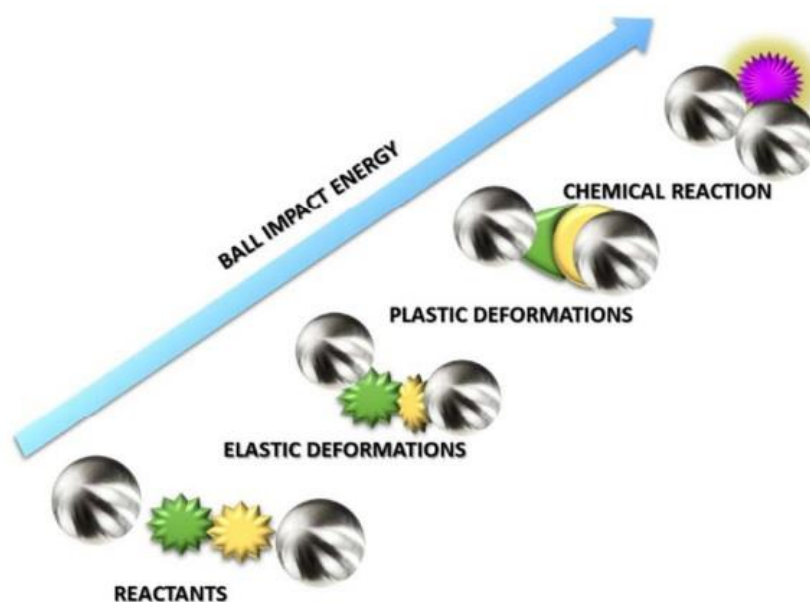


Figure 2.2 Reaction due to mechanochemical action [28]

The differentiating characteristic of mechano-chemistry is the ability to achieve chemical reactions either by dry milling or by wet milling under solvent-free circumstances. Thus, mechano-chemical methods are considered more efficient, environmentally friendly, and sustainable compared to conventional chemical reaction processes [36].

At laboratories and in industry, ball mills are widely utilized to reduce the particle size of many aluminosilicate materials such as cement and fly ash. The extensive use of ball mills, particularly in the construction industry, demonstrates the existence of various operational parameters that affect the mechanochemical activation process. The movement of the balls during the grinding process relies on different parameters of the

process. For instance, increasing the rotational speed or the feeding amount of the grinding chamber can reduce the efficiency of the process by influencing the energy transfer between the balls and the materials [37,38]. The overall impact energy tends to rise with increased rotational speeds; however, concurrently, the grinding balls tend to adhere and roll along the inner walls of the grinding chamber. Hence, this leads to a loss of impact or impact forces which means lower grinding efficiency at higher rotational speeds. [39,40]. Another factor which affects the efficiency of the grinding operation is the ratio of the grinding ball to the quantity of material to be treated. Increasing this ratio may increase the mass and kinetic energy of the system. This increases the probability of collisions and enhances the abrasion of the final product. Another effective parameter in the mechanochemical process is the grinding time. Thus, by ensuring an optimal grinding time, it is possible to achieve more reactive products with less energy [40].

There is a study on the utilization of fly ash left as waste in the environment as a building material. After examining parameters such as particle distribution, moisture content and density of fly ash, a laboratory type ball mill, vibrating mill and vertical mill were used to improve the existing reactivity of fly ash. The mechanical improvements of each mill on fly ash were observed. Geopolymer cylinder samples were obtained from the mechanically improved fly ash. XRD and Fourier Transform Infrared Spectroscopy (FTIR) results of fly ash and geopolymer samples were observed. The obtained data were used to test the 7-day compressive strength of the samples. In the ball mill, which was utilized, a grinding procedure of 10,20,30,60 and 120 minutes was applied respectively with a ball fill ratio of 30% and a material fill ratio of 1.1. In the vibrating mill, 70% ball fill rate and 1.1 material fill rate were used, and the material was ground for 10,20,30,60, and 120 minutes in the same manner. In the vertical mill, these times were performed as 1,2,3,4,5,7,10 minutes. According to the data obtained, more surface area was obtained in a shorter time with the vertical mill. In the study, it was observed that all grinding parameters did not affect the XRD graph pattern. In the FTIR results, there was no change in the peaks within the graph and only an increase in the intensity of the peaks due to the increase in surface area was observed. Although no differences were observed in FTIR and XRD results, changes in particle structures were observed in scanning electron microscopy (SEM) analysis. In addition, agglomeration was observed in the samples ground in a vertical mill for 10 minutes. According to the data obtained from compressive

strength tests, the highest compressive strength value was received in the sample ground in the vertical mill [41].

In a different investigation, kaolinite surfaces were modified through mechanochemical treatments lasting 10 hours. Results revealed a continuous reduction in the density of interval with increasing mechanochemical treatment duration, leading to the stratification of kaolinite and a subsequent reduction in crystallite size. Results indicated significant changes in the hydroxylation arrangements of kaolinite. Infrared spectroscopy tracking alterations in the structure of kaolinite hydroxyls showed a loss of hydroxyls after 10 hours of milling, evident in the decreased intensity of OH stress vibrations and deformation. Changes in the surface structure of the O-SiO units were captured in the SiO tensile. Simultaneously, the decline in the intensity attributed to kaolinite SiO₂ stretching vibrations corresponded with the increased intensity of extra bands, attributed to the newly synthesized kaolinite surface through mechanical processes. The mechanochemical treatment of kaolinite resulted in a transformed surface structure. [42].

A study observed the effect of nickel slag under different grinding parameters. The effect of grinding with mortar prepared with alkali activators on compressive strength values was observed. Additionally, the porosity structure of the system formed by mercury porosimeter was observed. To characterize the hydration products, thermal analysis, XRD analysis and FTIR test were performed on the specimens after hardening. The grain size of the raw nickel slag is between 3-4 mm. The size of the balls used in the mill were 30mm, 20mm and 10mm respectively. For the grinding process, equal amounts of all balls were used by mass. In addition, the ball filling rate varied between 30%, 40% and 50%, while the ball material ratio was determined as 10% and 15%. Grinding times ranged from 2, 4 and 6 hours. It is stated that high and low-ball material ratio decreases the efficiency of the grinding process. While long grinding times increase the fineness of the material, it is stated that it may cause agglomeration after a certain point. For the compressive strength test, samples were poured into 50x50x50 mm³ molds and test results were taken on the different days. Considering the strength results, compressive strength results are higher when the ball loading rate is 30%. Additionally, reduction in the ball filling ratio resulted in an increase in surface area and a decrease in the average particle

size. Increasing the ball material ratio also led to enhancements in surface area and average particle size. The optimum grinding time was found to be 4 hours according to the results and investigations. XRD analysis of the hydrated product revealed that an increase in the ball filling ratio and a decrease in the ball material ratio led to a reduction in C-S-H densities. This is evidence that hydration is less in these cases. Considering the values of the heat of hydration, the increase in the grinding time and the decrease in the ball loading ratio increase the heat of hydration. Increasing the grinding time and decreasing the ball filling ratio caused the porosity values to decrease. More C-S-H gels were formed with decreasing particle size and fineness and the voids in the system decreased, which led to a decrease in the porosity percentage. As the hydration reaction was completed over time, an increase in compressive strength results and a decrease in porosity were observed at later ages [43].

In another study, the research focused on examining the alterations in the surface of kaolinite induced by diverse thermal and mechanical operations. When subjected to heating within the range of 500 to 750°C, a substantial portion of the aluminum surface changed from octahedral to tetrahedral coordination, attributed to the process of dihydroxylation in kaolinite. Nevertheless, the findings indicated a distinct reduction in the Si/Al atomic ratio within the mechanically treated specimens, suggesting that the mechanical operation led to an increased concentration of aluminum on the kaolinite. Furthermore, the results highlighted that as the temperature increased (from 750 to 980°C), there was a thermal conversion to metakaolinite and mullite. [44].

In another article, it is studied the mechanical activation of bottom ash obtained from municipal waste disposal. A ball mill was used to perform the grinding process. The balls used in the grinding chamber have a ratio of 6:1 by volume to the ground material. Within the scope of the study, this volumetric ratio was kept constant, and the grinding times were 15, 30 and 45 minutes. The particle size distribution of the materials milled at different times was analyzed. Castings were made with waste ash, cement, and calcium carbonate to observe the effect of grinding parameters on compressive strength. The castings were made in 40mm x 40mm x 160mm molds and then cured for 7 and 28 days. The ground bottom ash samples and the mortar samples were tested by XRD, FTIR and

thermogravimetric analysis respectively. In addition, heavy metal leaching was also tested to observe the impact of the milled materials on the environment. According to the results obtained, decreases in the cumulative distribution curve values of the material were observed when the grinding time increased. In addition, an increase in surface areas was detected. XRD test results showed that the silicon content in the quartz structure in the unground material decreased by the grinding process. Thus, this increased the activity of the material. In FTIR analysis, an increase in the density of silicon oxygen and aluminum oxygen bonds was observed. These were attributed to the increased surface area and decreased particle size. It was determined that the highest-pressure value was obtained at 30 minutes grinding. At 45 minutes, agglomeration in the milled material was observed within the scope of SEM analysis. Taking this into consideration, it is better understood that the strength value of 45 minutes grinding time is lower than 30 minutes. When the FTIR analysis was performed on the grinded material, it was observed that the calcium hydroxide peak disappeared. It indicates that the consumption of calcium hydroxide in the system increases with the grinding time. Furthermore, the strength of silicon oxide and aluminum oxide bonds also decreased. This decrease is an indication that these structures are involved in the hydration reaction. When the results of the thermogravimetric analysis were analyzed, it was observed that the amount of calcium hydroxide was the lowest in the material grinded for 30 minutes. This is consistent with FTIR, XRD and compressive strength test results. As a result, with the help of the grinding process of waste bottom ash, the particle size decreased, and the material became more reactive [45].

A study investigated the amount of quartz content in the mechano-chemical kaolinite amorphization. Mechano-chemical activation (dry milling) deforms the crystal structure of kaolinite by breaking various bonds. The main mineral components of kaolin minerals are kaolinite and quartz. In this study, grinding experiments were carried out in a planetary mill for 1, 2, 3 and 4 hours. The grinding-induced disturbance and breakdown of the kaolinite structure are evident in the rise of average lattice deformation and the decline in peak regions. The escalation in quartz content led to a hastened amorphization of the kaolinite structure induced by mechanical process. After 4 hours of grinding, the kaolinite's crystalline structure was entirely disrupted in the sample containing 75 wt%

quartz. The findings indicate that quartz grains function as milling elements in the process of intensive dry milling of kaolinite [46].

In a different investigation, combinations of two waste materials were employed to create cementitious materials through the process of mechanochemical activation. The activation was performed using abrasive mill grinding. Due to the impact, the absorbed energy may have led to alterations in the particle surfaces, causing dislocations, point defects, and other structural irregularities. This results in materials possessing enhanced free surface energy, rendering them more reactive. The findings indicate that individually grinding the two materials is the most efficient approach for activating them, yielding optimal paste properties [47].

It is used construction demolition waste which contains more than 50% silicon in another research. A ball mill was used for the grinding process. The effect of different grinding parameters on the reactivity of construction demolition waste was observed. The grinding parameters were defined as 879 cm³ total ball volume, 192 cm³ material feed, 1,2,3 and 4 hours grinding time. The construction demolition waste was firstly passed through the jaw crusher and then subjected to the grinding process. Alkaline activator prepared with sodium hydroxide and sodium silicate was used for geopolymer synthesis. The geopolymer mortar poured into 50x50x50 mm³ molds and cured at 50 degrees Celsius for 7 and 28 days. Particle size, solubility, amount of reactive phase by Rietveld method, XRD analysis, FTIR analysis, isothermal calorimetry analysis, XPS analysis, SEM analysis and compressive strength test were analyzed. According to the data obtained in particle sizes, it was observed that agglomeration increased after 4 hours. In addition, it was observed in 3D laser microscopy that after 4 hours the milled material had a larger and irregular particle structure. In the reactive phase results, it was observed that the grinding time significantly increased the aluminosilicate content. The results revealed that quartz, calcite, and albite crystal structures were observed according to XRD analysis. The increase in grinding time caused a decrease in the densities of these crystal structures. Especially a significant decrease in albite density was recorded. According to the compressive strength results, the increase in grinding time positively affected the strength achievements. As a result of the study, it was observed that the grinding process affects

the reactivity of construction demolition waste by changing the particle size, crystal structure and surface group. In addition, Al and Si solubilities improved by increasing the amorphous structure. The grinding process increased the Si/Al ratio in the structure and increased the silicon-rich gel formation because of the reaction. Thus, this situation caused an increase in compressive strength [48].

In a different investigation, the study explored the improvement of reactivity in the fine size fraction of construction demolition waste through mechanical activation by stirred mill. Systematic measurements were conducted to gather insights into reactivity. Alterations in particle shape and surface were assessed using SEM, while structural changes were examined through FTIR and XRD analyses. The impact of mechanochemical activation on pozzolanic reactivity was evaluated through lime consumption and durability tests. The research concluded that the reactivity of the fine size fraction of construction demolition waste can be adjusted through the process of mechanochemical activation [49].

2.3 Effects of Different Grinding Parameters on Construction Demolition Wastes and Various Materials

There have been studies on the grinding of construction demolition waste, but these studies have generally focused on determining the utilization ratio of the waste in the system rather than determining the optimization of the grinding parameters. In a study, replacing 20% of the cement with fine waste resulted in a reduction in the 7-day compressive strength about 25%. As the replacement percentage increased, the reduction effect of the admixture became more significant on the strength [50]. In another study, ground silica-rich ceramic below 75 μm was replaced with cement. 20% replacement reduced the compressive strength by about 20% and 15% at 7 and 28 days, respectively. In addition, the flow value of the test mixes decreased compared to the control mix with 100% cement [51]. It is investigated the substitution of waste brick dust into cement in another research paper. They concluded that at constant w/b ratio, workability decreased as the content of waste admixture increased. Moreover, more than 10% replacement dramatically reduced the compressive strength at all ages [52].

Another study investigated the usability of cement with a mixture of ground construction demolition waste. Construction demolition waste which is similar to the particle size distribution of normal Portland cement was used. A slight reduction in the flow of all test mixtures was observed, which was attributed to the higher water absorption of the ground brick dust. Furthermore, a replacement rate above 18% decrease the compressive strength, while a smaller replacement only showed a filling effect and no noticeable pozzolanic activity [53]. It is studied the effect of ground brick powder on fresh and hardened concrete properties in another study. It is found that workability decreased when 10-30% of cement was replaced by brick. The compressive strength decreased significantly at early age (<28 days) by the addition of brick dust, while a 10% reduction at 90 days showed similar strength to the reference mix. The loss of strength was attributed to the low reactivity of the brick powder, and it was explained that finer ground brick powder would perform pozzolanic reaction and provide higher strength [54]. In another article, it is studied the effect of recycled aggregate and ground brick dust on the mechanical properties of concrete. They found that finely ground brick dust particles increased the pore distribution of the matrix and increased the density [55].

It is investigated the recycling of waste ceramics in another research. Various combinations of cement-ceramic blends were formulated by incorporating up to 30% by weight of recycled waste material. The mineralogy of the powdered ceramics was checked by XRD analysis and FTIR spectroscopy. After the materials were completely characterized, lime consumption and Frattini tests were conducted. The FTIR results revealed that the reduction in crystallinity was associated with the activity of the calcined clays. This research reveals that ceramic wastes which is calcined at 950 °C can be used as very effective pozzolans by appropriate fine grinding. The activation is attributed to crystallochemical changes that occur during calcination. The calcination process led to significant disturbance in the crystalline structure of the clay material. Consequently, the interaction with CH and the utilization of portlandite were detected through XRD analysis after 28 days of hydration. [56].

A research study investigated the impact of grinding on the physical characteristics of F-Grade fly ash and clinker. The fineness of fly ash demonstrated an increase with

prolonged grinding time, but this increase becoming less noticeable after 2 hours. Grinding also brought about alterations in the morphology of the fly ash, breaking down most spherules and large irregularly shaped pieces after 2 hours of grinding. The optimal milling duration for fly ashes appeared to be around 4 hours. Beyond this duration, the water demand increased, and the strength activity index either declined or exhibited no significant improvement. [57].

In another study, fine grinding of silica fume (SF) was considered as a method to improve the effects and performance of coarse SF in the production of high durability. The coarse SF, characterized by a 45 μm sieve residue of around 32%, displayed limited pozzolanic reaction. To enhance its property, the coarse SF underwent grinding until the average particle size was decreased, resulting in 45 μm sieve residues of 4% and 1%. Subsequently, it was blended with Portland cement at a weight ratio of 25%, and the strength activity index was assessed for the production of high performance concrete. The pozzolanic reactivity of SF in the composite cement system and its quantitative effect on mechanical strength were studied in detail. X-ray fluorescence (XRF) data results showed that the reactive SiO_2 content had a significant effect on pozzolanic properties, but not as significant as the SF particle size [58].

It is aimed to investigate the grinding criterions for the recycled materials such as marble, tile, brick and phosphogypsum in another research. The recycled materials were evaluated by grinding in a planetary ball mill at various grinding duration and rotational speed. The tailings were characterized by differential scanning calorimetry (DSC), BET specific surface area, XRF, TGA and XRD. The specific grinding energy requirements were linked to the mineral composition of the phases, leading to lower grindability indexes for wastes which were formed at higher temperatures. Brick and clay tile waste were found to have the capability to be utilized as supplementary cementitious, but clay tile waste required more energy during grinding processes. Wastes characterized by mineral compositions developed at elevated temperatures exhibited increased specific grinding energy requirements and decreased grindability indexes. Wastes with mineral compositions formed at higher temperatures have higher specific milling energy demands [59].

The objective is to examine the impact of 400 days on the microstructure, durability, and mechanical properties of mortars with the addition of waste brick dust with 20% cement clinker replacement in another research. As per the findings, mortars incorporating 10% and 20% brick dust demonstrated favorable long-term service properties and outperformed ordinary Portland cement mix. The microstructural analysis conducted in this study validated the pozzolanic reactivity of the waste brick dust. [60].

Another study showed an extensive research approach for the utilization of recycled aggregates from brick structures that had been demolished. The masonry waste was pulverized, categorized, and identified as coarse, fine, and powdered (CBP). The initial stage of the study focused on how recycled aggregates influenced the physicochemical properties. Non-traditional tests were performed, including XRD, TGA, and microstructural analysis. The program's second phase evaluated the impact of employing recycled aggregates on the properties of concrete. The findings indicated that the cement paste, altered by incorporating 25% CBP, demonstrated a reduction in pore size and experienced lower weight loss when exposed to high temperatures compared to the reference paste [61].

Another research is aimed to investigate the possibility of partial cement replacement of container glass, brick, and tile waste from the red clay ceramic industry. Various forms of waste were grinded to specific sizes and their pozzolanic activity was analyzed. The reduction in size of waste particles was conducted through crushing and grinding, utilizing a laboratory ball mill. Following the grinding process, the fine waste materials underwent characterization, including density determination, analysis of Blaine fine granularity, and examination of particle morphology using scanning electron microscopy. Furthermore, each finely ground waste was substituted into cement with ceramic and the strength activity index was determined at 7, 28 and 90 days. The results obtained confirmed the pozzolanic activity of the milled wastes and demonstrated their feasibility of integration into cement mortars and concrete components [62].

In another research, mine waste is grinded at different time and observed the effect of grinding on the pozzolanic property of the material. A laboratory type ball mill was used.

The ball sizes were specified as 5, 8 and 15 mm and the mass ratios were 3:5:2, respectively. The mill with 500 mL grinding chamber was fed with 80 g of material in each grinding process. The total ball mass was 600 g and steel ball type were used. The material was milled separately for 5, 20, 40, 80, 120 and 160 minutes. As a result of the grinding processes, the D50 value of the material milled for 80 minutes is 1.45 μm . Beyond 80 minutes, there was no significant decrease in particle size. It was stated that this was due to the agglomeration of the material particles at longer grinding times. It is stated that mechanical grinding causes a decrease in particle size and relative crystalline structure. It is stated that the increase in the surface area, pore volume and disruption of the lattice structure of the material increases the pozzolanic activity. In addition, it is reported that the grinded quartz structure in the material reacts with calcium hydroxide and therefore forms C-S-H gels [63].

To provide a theoretical background for the utilization of mine wastes as cement additives, another study investigated the pozzolanic reactivity of muscovite before and after mechanical activation process. The study revealed that muscovite exhibited significant pozzolanic activity because of structural and morphological transformation caused by mechanical grinding. The muscovite that has been activated after 160 minutes of mechanical activation meets the requirements for use as a supplementary cement additive. The main characteristics of this pozzolan are: 11.7 μm median particle size (D50), 28.82 m^2/g BET specific surface area, 14.99% crystallization and 94.36% pozzolanic activity index. Continuous grinding resulted in a progressive reduction in relative crystallization and an elevation in the pozzolanic activity index, attributed to the dehydroxylation reaction induced by mechanical activation. Mechanically activated muscovite demonstrated its capacity to interact with CH, a characteristic feature typical of pozzolans. [64].

Comprehensive models based on particle size distribution were employed to predict the particle sizes of grinded clay bricks (GCB). These models encompass variations in grinding times (20 minutes, 40 minutes, 60 minutes, 80 minutes, and 100 minutes) during the milling of crushed material. The impact of prolonged grinding time on the milling of material was examined to establish an approximate correlation between GCB particle

sizes and grinding time. Ball mill grinding was carried out at 20 to 25°C using 30 mm diameter steel balls at a grinding speed of 1260 rpm. The crushed material was grinded for 20, 40, 60, 80 and 100 minutes. The 8 kg sample mass was kept constant during all ball grinding durations. Grinding duration was observed to have a direct effect on GCB particle sizes and clay brick powder (CBP) content. Particle size distribution results revealed that increases in grinding times decreased CBP particle sizes [65].

Another study examined the impact on particle size and structure of composite fine material produced through high-energy ball milling. In this investigation, fines of tungsten and copper were subjected to grinding in a planetary mill for varying durations. The ball to powder weight proportion was set at 10:1, and three different grinding speeds (200, 300, and 400 rpm) were applied during the experiments. A 2 wt% stearic acid was utilized as an agent to prevent total cohesion and reduce the tendency for cold welding between powder particles. The structural changes of the ground fines were assessed through SEM. Additionally, the study determined the variation in particle size and fine morphology based on the grinding period. The findings indicated a substantial influence of grinding duration on the structural transformation of the powder, with the optimum particle size being dependent on the grinding speed [66].

Lately, there has been substantial discourse in the literature regarding the correlation between roughness of surface and the fine granular structure of materials. The significance of utilizing surface roughness in pozzolans has been established for cement based mixtures. Despite previous research efforts to assess the impact of grinding processes on the surface roughness of pozzolanic materials, there has been a scarcity of dedicated studies employing multiscale characterization methods to analyze this intricate parameter. In this study, an innovative and cost-effective technique was employed to characterize the surface roughness of clay brick fine undergoing various grinding processes with various specimens. The primary aim of this research was to explore the connections between fine granule sizes and surface roughness parameters of crushed clay bricks. Crushed masonry bricks were ground using a ball mill. The sample masses employed for the grinding process were 8-12 kg. A consistent grinding time of 60 minutes was maintained for all sample masses. The grinding speed applied to all sample masses

was set at 1260 rpm. The characterization of clay brick powder obtained from the ball mill was carried out using scanning electron microscopy. Tests to find significant trends in surface roughness parameters between grinding process were not successful. The outcomes from amplitude roughness parameters, transforms, autocorrelation functions, and power spectral density functions did not exhibit notable patterns for clay brick powder samples ground under varying sample masses. The surface roughness characterization algorithms used in this study are considered to contribute to the understanding of the surface roughness properties of clay brick powder samples subjected to different levels of fine particles [67].

In another research, tests such as Frattini test, lime consumption, electrical conductivity, and Strength Activity Index (SAI) are utilized to determine the pozzolanic activity of calcined clay. They found that the Frattini test (direct) correlates well with SAI (indirect) and could be used more specifically in the future. Furthermore, the electrical conductivity test is also an indirect method of determining the pozzolanic activity of a material, which is based on the change in conductivity due to pozzolan suspensions in CH solution. However, there is a lack of an adequate number of methods used to evaluate pozzolanic activity. The findings of one pozzolanic activity test cannot be easily confirmed by other methods. Usually, SAI cannot be easily verified by chemical methods. In addition, the filler effect can be confused with pozzolanic activity in the results of SAI. Therefore, verification of pozzolanic activity by mechanical, chemical and physicochemical methods is very beneficial. Furthermore, SAI is a relatively slow method for the assessment of pozzolanic activity. The development of other faster mechanical tests is important for the simplicity of future research [68].

Another study investigated alternative methods of changing the grinding media size distribution and feed material particle size distribution to improve the grinding efficiency of a laboratory-scale ball mill.

Silica ore served as the test material in this study. The consistent experimental parameters included grinding media feeding, powder feeding, and rotational speed of mill. The data gathered from the experiments underwent analysis through a model-free approach known

as the Attainable Region method. This analytical method revealed that achieving the desired product fineness is contingent on the distributions of grinding media type and size of feeding material. Furthermore, the experimental results showed the requirement to establish a correlation between ball size and feed size distributions to enhance the grinding efficiency of a ball mill and to ensure optimal production of material of the required size [69].

In another investigation, the influence of ball diameter sizes on the grinding process was explored. A laboratory-scale ball mill was employed, utilizing ball media with diameters of 10-30 mm. Quartz was chosen as the experimental material and was organized into three mono-sizes. A milling study was also carried out which included a mixture of the three ball diameters. The results showed that among the three sizes, balls with a diameter of 30 mm were the most effective. The balls with a diameter of 10 mm were identified as the least efficient, as they showed minimal particle breakage, whereas the balls with a diameter of 20 mm were effective up to a certain extent. However, a milling operation with different ball diameters performed slightly better than the 30 mm diameter ball operation. It was observed that the primary breakage function was not related to the ball diameter, but it is related to the feed material [70].

Study on the pozzolanic activity of clay brick waste in Nanjing region by using a ball mill is conducted in another research. Within the scope of the research, waste materials were grinded at different times. Studies were carried out on the activity of wastes milled for 10, 30, 60, 120 minutes. The D50 value of the material grinded for 120 minutes was 3.4 μm , the D50 value of the material grinded for 60 minutes was 10.5 μm , the D50 value of the material grinded for 30 minutes was 15.8 μm and the D50 value of the material grinded for 10 minutes was 27.1 μm . The increase in grinding time significantly reduced the particle size of the material. In addition, the surface area of the materials increased due to longer grinding time. Another analysis performed was the castings made with a cementitious system. XRD data of 28 and 90-days hydrated mortars made with 30% by mass substitution of cement were obtained. According to the results, it was stated that the grinded material reacted with calcium hydroxide, which is the hydration product of cement, and formed C-(A)-S-H gels [71].

In another study, it is described the pozzolanic properties of pastes containing waste bricks from construction and demolition waste. Waste bricks were grinded until all particles pass through #200 mesh sieve. According to the experimental results, mixtures where waste brick waste is substituted with cement at a rate of 10% yielded better long-term strengths compared to the reference mixture. Thus, increasing densification was obtained by decreasing the amount of CH. These findings emphasized the potential of waste brick as a cement replacement material [72].

3. MATERIALS AND METHODOLOGY

3.1 Materials

Materials which are used for test methodologies are categorized and defined in this section. Ordinary Portland cement, mix brick waste, which is collected from construction demolition, standard sand which is met the requirement of BS EN 196-1 and CH is utilized to design mortar and paste mixture.

3.1.1 Characterization of CDW Materials

CDW materials which are RT, HB and RCB were utilized for grinding process. All wastes were collected from different demolition sites in Eskisehir. RT consists of materials from the roofs of demolished buildings, while HB was collected mostly from demolished walls. In addition, RCB was collected from the front facade of the building. The collected waste materials were transferred to the laboratory and classified. The different raw materials were first crushed into relatively smaller pieces with a hammer before being fed into the jaw crusher which is shown in Figure 3.1.



Figure 3.1 Laboratory type jaw-crusher which is utilized to decrease the maximum grain size to 2mm

Each material was then fed into the jaw crusher separately and crushed to a maximum grain size of 2.00 mm. Maximum grain size controlled by sieving the crushed material. Residue materials were fed into the jaw crusher again for representative crushing. The crushed materials were stored in different buckets.

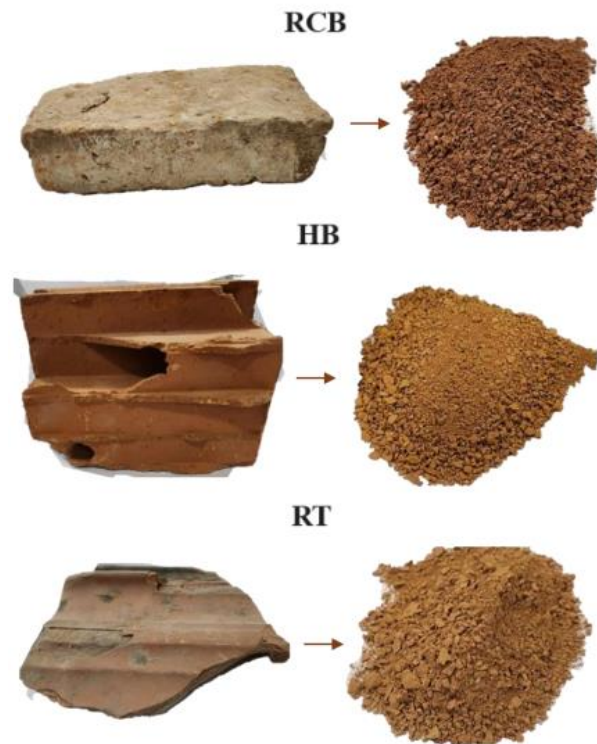


Figure 3.2 CDW precursor stages from raw to crushed material

Each waste with a maximum grain size of 2mm was kept in a 100 °C oven for one day to eliminate the moisture in it. Density calculation for each type of oven dry waste was performed with a pycnometer. The pycnometer method is a technique to calculate a material's density. A pycnometer, which is an individual glass or metal container with an exact volume, is used in this procedure. In the density measurement, the empty volume of the pycnometer was first measured and recorded as M_1 . The total mass of the pycnometer and the material was labeled M_2 and the total mass of the pycnometer, material and water at constant volume was labeled M_3 . Finally, the total mass of only the pycnometer and water at constant volume was labeled M_4 . The density of each material was calculated separately by following equation:

$$\rho = \frac{M_2 - M_1}{(M_2 - M_1) - (M_3 - M_4)}$$

X-ray Fluorescence is an analytical technique that measures the elemental composition of a substance by interacting with X-rays. XRF is a rapid and non-destructive method. Furthermore, XRF is suitable for solids, liquids, and powders in most cases. XRF spectroscopy is an efficient approach for qualitative and quantitative material composition studies. When X-rays are released on a material, some of the X-rays move through the material while others are absorbed. The absorbed X-rays interact with the material at the atomic level. Thus, it causes a variety of events including scattering and releasing photons, electrons, and fluorescent X-rays [73–76]. Before XRF measurement, sample preparation took place. For XRF test, there are several methods to prepare dry sample. First method is initiated by pouring powder into sample cup, second approach is initiated by pellet sample preparation. For second approach bottom of powder is covered by wax material such as boric acid (H_3BO_3) after this process these two materials are pressed, and pellet sample obtained. Usually powder to wax binder ratio is 10:1 or 10:2.

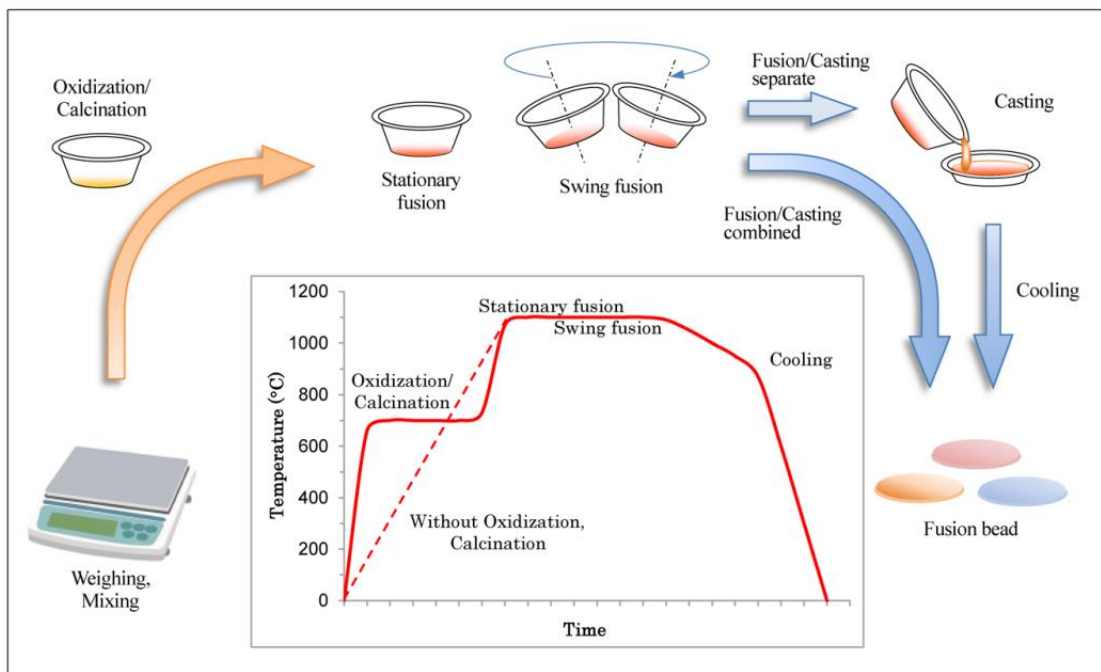


Figure 3.3 Fusion bead preparation procedure [77]

The last one which is third approach is fusion method. In this approach, certain amount of powder specimen and flux such as lithium tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$) are mixed in the crucible. Afterwards, mixed materials are fused in the oven at 1000-1200 °C. Crucible left stationary in the oven until the specimen melt. To remove bubbles inside the melted material and homogenize the mix, crucial is swinged. For cooling proses, melted material poured into a plate and left until cool down to receive fusion beads. Fusion beads are used for XRF measurements to obtain elemental information of the sample. Fusion method provides more accurate result due to homogenized form and reduced physical effects of material such as grain size, mineralogical effect [77,78]. In this research third method which is fusion is used to determine the elemental content of waste materials. Spectro XEPOS 03 STD instrument was used for the XRF analysis.

Table 3.1 Elemental content of CDW based materials

Symbol	Concentration (%)		
	HB	RCB	RT
SiO₂	60.23	59.92	53.13
Al₂O₃	18.69	19.91	16.17
Fe₂O₃	7.42	7.51	11
CaO	3.58	2.95	7.53
MgO	2.47	2.45	5.85
SO₃	0.94	0.6	0.45
Na₂O	0.28	0.26	0.27
K₂O	3.45	3.63	2.03
TiO₂	0.91	0.99	1.69
Cr₂O₃	0.03	0.04	0.06
Mn₂O₃	0.13	0.13	0.19
P₂O₅	0.24	0.29	0.32
ZnO	0.01	0.01	0.01
Loss of Ignition (%)	1.51	1.3	1.21
Specific Gravity	2.78	2.6	2.71

When the values in Table 3.1 are analyzed, it is seen that 60.23% of HB content, 59.92% of RCB content and 53.13% of RT content are SiO₂. Thus, it is understood that the major element in all three wastes is silica. Aluminum content, which is between 16-20% in all

three wastes, is the second highest oxide by percentage. The amounts of calcium oxide and other oxides are less than 10% for all three wastes. It is obvious that all brick wastes have similar elemental content as seen in Table 3.1. Therefore, HB, RCH and RT wastes were mixed homogenously in equal weight after crushing process.

Table 3.2 Elemental content of mixed CDW based materials

Symbol	Concentration (%)
	Mixed Brick Waste
SiO₂	60.06
Al₂O₃	20.28
Fe₂O₃	7.42
CaO	1.94
MgO	2.75
SO₃	0.94
Na₂O	0.02
K₂O	3.61
TiO₂	0.98
Cr₂O₃	0.02
Mn₂O₃	0.13
P₂O₅	0.18
ZnO	0.01
Loss of Ignition (%)	1.44
Specific Gravity	2.7

Major element is SiO₂ in mixed brick waste as can be seen in Table 3.2. Mixed waste has 60.02% SiO₂, 20.28% Al₂O₃ and 7.42% Fe₂O₃ content. There are also some elements in lower amount such as % 1.94 CaO, 2.75% MgO and 3.61% K₂O. This elemental analysis of mixed brick represents average chemical properties of different bricks. In this study, all test methodologies are carried out by utilizing mixed brick waste.

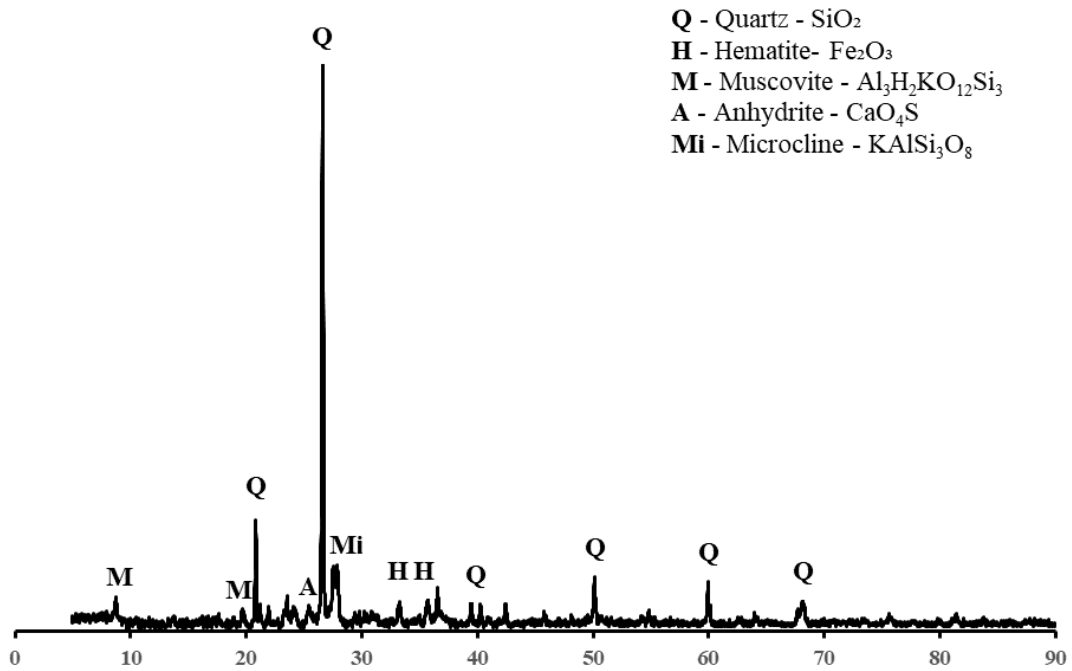


Figure 3.4 XRD pattern of mixed brick waste

Molecular structures of homogenously mixed brick waste are analyzed by XRD as shown in Figure 3.4. XRD data is obtained between 5 and 90°. Data result of XRD analysis shows that mixed brick waste has intense Quartz peak of SiO_2 . Thus, mixed brick waste is mainly composed of Quartz. Additionally, there are several peak patterns which are Muscovite and Hematite in different angles. Minor peak patterns such as Anhydrite and Microcline are also revealed in the graph.

3.1.2 Standard Sand

Sand meeting BS EN 196-1 standard was supplied for each casting to have a uniform and controllable sand content in the mix design of the castings to be made within the scope of the research. It is a natural, siliceous sand which is containing rounded particles and a silica content of at least 98%. In addition, moisture content of standard sand should not exceed 0.2% by mass [79].

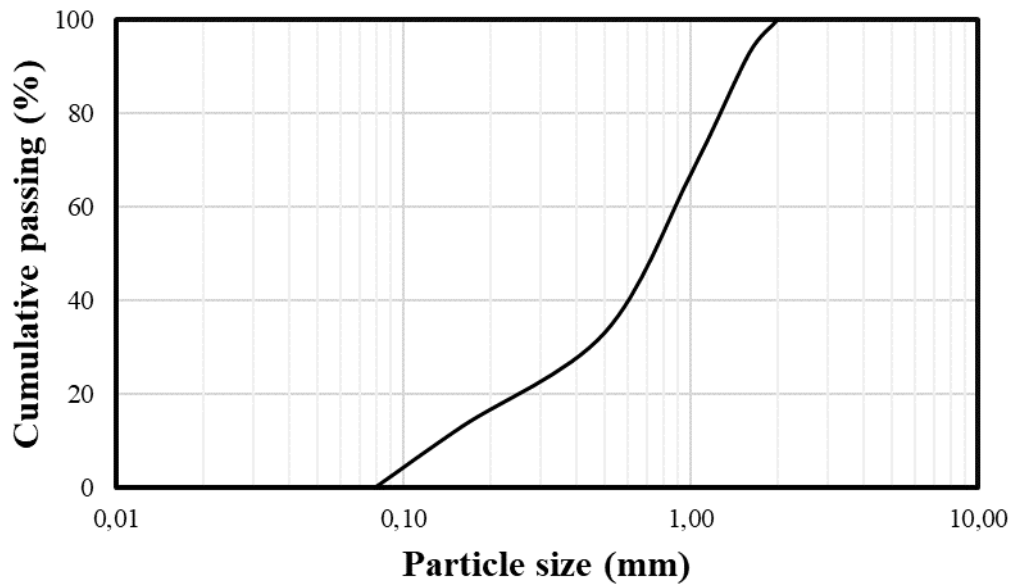


Figure 3.5 Particle size distribution of standard sand

3.1.3 Calcium Hydroxide (CH)

Calcium hydroxide is a white odorless material. It has low solubility in water (1.2g/l at 25 °C). In addition, the solubility of calcium hydroxide reduces with increasing temperature. Calcium hydroxide is an alkali with a pH around 12.5-12.8 [80]. Aluminum and silica-containing materials with high surface area can form binding systems in the presence of alkali. Calcium hydroxide dissociates into Ca^{+2} and OH^- ions in the presence of water. Ca^{+2} free ions in the matrix react with SiO_2 and Al_2O_3 in the system. Thus, C-S-H and C-A-H hydrate gels that provide strength are produced [81,82]. Calcium hydroxide with 87% purity was used in the study.



Figure 3.6 Calcium hydroxide which is used in the study

3.1.4 Ordinary Portland Cement

Ordinary Portland cement is obtained by fine grinding of clinker produced in cement plants by specific grinding processes. At least 2/3 of the compounds in Portland cement clinker should consist of tricalcium silicate (C₃S) and dicalcium silicate (C₂S) which are classified as calcium silicate. Cement is an inorganic hydraulic material that sets and hardens by reacting with water (hydration) and maintains its durability for a long time. CEM I type Portland cement is a cement that does not contain blast furnace slag, silica fume, pozzolan, fly ash or limestone. It contains 95% clinker and 5% additional minor components [83]. In this study, CEM I 42.5 R cement type was used in order to observe the binding properties of the construction demolition waste as well as to develop a stable test methodology.

Table 3.3 Elemental content of ordinary Portland cement

Symbol	Concentration (%)
	CEM I
SiO ₂	19.93
Al ₂ O ₃	5.61
Fe ₂ O ₃	3.29
CaO	64.32
MgO	1.22
SO ₃	2.71
Na ₂ O	0.21
K ₂ O	0.78
TiO ₂	0.23
Cr ₂ O ₃	0.03
MnO	0.13
P ₂ O ₅	0.48
ZnO	0.01
Loss of Ignition (%)	1.5
Specific Gravity	3.15

3.2 Methodology

Details of sampling method to provide representative sample from CDW based material is explained. In addition, type of grinding equipment, type, size and amount of grinding

media, calculation method of MFR and BFR and lastly determined grinding parameters are detailed in this section.

3.2.1 Sampling

Large quantities of samples are more representative of the total supply. In addition, to perform laboratory tests, large quantities of samples may need to be reduced to quantities that can be used for laboratory testing. However, since the large quantities of sample available have a specific gradation, the sample divided into relatively smaller pieces should also have a corresponding gradation. For this reason, the samples to be used on a small scale should be representative of the total supply [84]. Within the scope of this study, the quartering method in ASTM C702 standard was used to allow large quantities of specimens to be utilized in the laboratory test procedures. Firstly, all material is placed on a clean flat surface. Afterwards, the sample is flipped over 3 times to completely mix the material. While the material is being mixed, each shovelful of material is poured from the top to form conical pile. The material is flattened with shovel to fixed thickness. The flattened material is carefully divided into four equal parts and the samples in the opposite corner are separated. The remaining two parts are mixed again, and the same methodology is continued until the desired quantity is obtained.

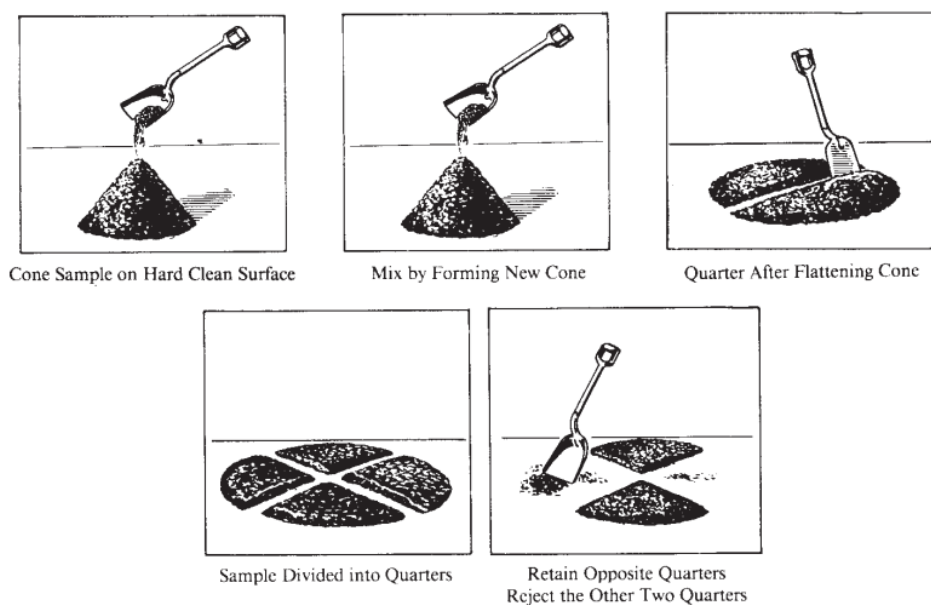


Figure 3.7 Steps of quartering method to obtain representative test samples [84]

3.2.2 Grinding Process

In this section, detailed information about grinding equipment and grinding parameters is provided. Selection of grinding equipment type and grinding media size, amount is indicated. Parameters divided into two main categories: MFR and grinding duration. Calculation of grinding parameters which was carried out for grinding process of waste material were defined in detail.

3.2.2.1 Grinding Equipment

In the context of this study, a laboratory type ball mill in accordance with TS 7700 standard was used to perform the grinding process as seen in Figure 3.8. The radius of the mill chamber is 15.25cm and the depth is 30.5cm. The mill volume is approximately 22 liters. In addition, the rotation speed of the mill is 70 revolutions per minute.



Figure 3.8 Laboratory type ball mill which is used to grind brick waste material

Steel balls of different sizes were used in the chamber to perform the grinding process. In the research, different quantities of five different types of ball sizes were utilized. The diameters of the ball sizes are 38.1, 31.75, 25.4, 19.05, and 15.87 mm respectively. A total of 43 balls of 38.1 mm diameter, 67 balls of 31.75 mm diameter, 10 balls of 25.4

mm diameter, 71 balls of 19.05 mm diameter and 94 balls of 15.87 mm diameter were selected as shown in Table 3.4. Average ball density calculated as 7.77 gr/cm³. Grinding was carried out for the desired time with the digital timer which is placed on the mill [85].

Table 3.4 Diameters and quantities of ball

Diameter of Ball (mm)	Amount
38.1	43
31.75	67
25.4	10
19.05	71
15.87	94
Total:	285

3.2.2.2 Determination of Grinding Parameters

Clinker and mineral additives produced in the cement industry must be grinded to a certain fineness to provide the desired quality. Grinding parameters should be optimized to reach the required fineness values. Among the most significant parameters affecting the efficiency of the grinding process are the type of mill used, ball fill rate, material fill rate and grinding time [86].

A cascading motion is evident, where the balls are attracted by the milling chamber wall and subsequently roll over each other from the upper to the lower part of the stack. At this filling rate, there is no segregation of the balls from the milling chamber wall or other grinding media. Consequently, each ball maintains constant contact with another grinding media or the milling chamber wall. Under these conditions, the primary grinding mechanism on the material is predominantly compression and shear stress occurring between the balls or between the grinding media and the milling chamber surface. A pattern of movement known as cataracting develops as the filling rate changes. Some of the grinding medias separate from the grinding chamber surface and take a parabola trajectory before hitting the ball accumulation. In addition to stress through compression and shear, there is also stress through impact. As the ball fill rate is increased further, the stack occupies so much of the space inside the milling chamber that some of the balls are

centrifuged. The balls on the chamber wall rotate without relative speed [87]. The filling rate of the grinding chamber affects the movement of the balls. The grinding performance obtained also changes with the changing ball movement. As the material filling rate increases, the stress of the balls on the material reduces and the effectiveness of the grinding process declines [88]. The grinding time, which is another parameter affecting the grinding performance, may cause agglomeration effect on the material after a certain period. As the grinding time increases, an increase in the fineness of the material is observed. In addition, increases in reaction rate were also observed with the increase in fineness. Therefore, it is essential to optimize the grinding time [89].

In this study, the effect of material fill ratio and grinding time as different grinding parameters were observed. The calculation of each grinding parameter was performed with a constant ball filling ratio.

The information obtained from the literature research shows the significance of the effect of BFR, MFR and grinding time on the grinding process. Therefore, to realize the grinding process, it is required to calculate the BFR, MFR and determine grinding time. The volume of the chamber and the areas of the balls used in the chamber were calculated. Figure 3.9 shows the cross section with the chamber filled with balls.

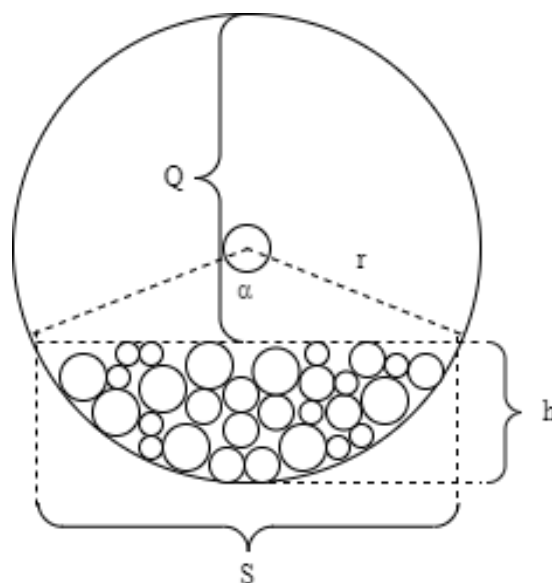


Figure 3.9 Cross-section of the chamber with grinding balls

The following formula was used to calculate the BFR and MFR:

$$\begin{aligned}
 S &= \text{Sin} \left(\frac{\alpha}{2} \right) * 2 * r \\
 Q &= \text{Cos} \left(\frac{\alpha}{2} \right) * r + r \\
 h &= 2 * r - Q \\
 \text{Filled Area} &= \left(\frac{\alpha}{360} * \pi * r^2 \right) - (r - h) * \frac{S}{2} \\
 \text{Total Area} &= \left(\frac{\alpha}{360} * \pi * r^2 \right) \\
 \text{Ball Filling Ratio (BFR)} &= \frac{\text{Filled Area}}{\text{Total Area}}
 \end{aligned}$$

After the chamber was filled with the ball, the S value in the equation above was measured to make the required calculations. As a result of the measurement, the S value was measured as 26.8 cm. By using the S value in the equations, the values of other unknown parameters were found theoretically as in Table 3.5.

Table 3.5 Parameter values of the mill

Parameters	
r	15.25 cm
S	26.8 cm
α	123°
Q	22.53 cm
h	7.97 cm
Filled Area	152.11 cm ³
Total Area	730.62 cm ³
BFR	~20%

For the calculation of the grinding parameter different ball types were used as mentioned in “Grinding Equipment” section. The filled area, which represents approximately 20% of the total area, indicates the volume of the balls and the spaces between the balls. The volume of the balls was subtracted from the filled area to calculate the volume between the balls. The filling ratio of the remaining empty volume is defined as MFR. The

theoretical calculation indicates that approximately 60% of the total filled volume corresponds to the ball volume, while the remaining 40% represents the volume of the space between the balls as shown in Table 3.6.

Table 3.6 Empty space volume percentage

Theoretical Total Ball Volume	2913 cm ³
Total Ball Volume for Cross-section	96 cm ³
Total Filled Volume for Cross-section	152.11 cm ³
Ball Volume Percentage	~60%
Empty Space Volume Percentage	~40%

The obtained data demonstrates that 20% of the total chamber volume represents the ball and space volume. 40% of this volume represents the space between the balls. The filling ratio of the space between the balls defines the MFR value. MFR ratios of 60%, 80%, 100% and 120% were selected for this study. The different MFR ratios were milled for 60 minutes and the optimum MFR ratio was determined. For the subsequent grinding process, grinding was carried out with different grinding times at the optimum MFR ratio. The quantities of mixed brick waste that will be grinded for specific MFR ratios are shown in Table 3.7.

Table 3.7 Amount of mix brick waste to feed into the mill

Chamber Volume	22284 cm ³
Total Volume of %20 BFR	4457 cm ³
Empty Space Volume of %20 BFR	1783 cm ³
%60 MFR	2888 g
%80 MFR	3851 g
%100 MFR	4813 g
%120 MFR	5776 g

After determining the optimum MFR ratio, the effect of grinding time was observed. The grinding times were selected as 30, 60, 120 and 180 minutes. The grinding parameters are shown in Table 3.8.

Table 3.8 Grinding parameters

Specimen ID	
M120-T60	20% BFR - 120% MFR - 60 minutes
M100-T60	20% BFR - 100% MFR - 60 minutes
M80-T60	20% BFR - 80% MFR - 60 minutes
M60-T60	20% BFR - 60% MFR - 60 minutes
M80-T30	20% BFR - 80% MFR - 30 minutes
M80-T120	20% BFR - 80% MFR - 120 minutes
M80-T180	20% BFR - 80% MFR - 180 minutes

3.3 Mixture Preparation

Two different mortar mix designs which are cement-based and waste-based is detailed in this section. Different mixtures were prepared for each grinded brick waste material to observe grinding effect on compressive strength.

3.3.1 Cement-Based Systems

The cement mortar mix for the investigation of pozzolanic activity was prepared. The mixture content consists of cement, pozzolanic material and standard sand as shown in Table 3.9. The water/binder ratio was fixed to 0.5 within the scope of the study. Mixed brick waste was added as pozzolanic material in the mix. Mixtures were prepared with each grinded material to observe the effect of the grinded material under different grinding parameters. The reference mix was prepared with 100% cement content. In addition, 20% of the total cement content in the reference mix was substituted with mixed brick waste. Specimens were cured for 7 and 28 days to test compressive strength [90].

Table 3.9 Mixture content of cement-based systems

Specimen ID	Aggregate (g)	Water/Binder	Dry Mixture (g)	
			Cement	Mixed Brick Waste
C-R	1375	0.5	500	-
C-M120-T60	1375	0.5	400	100
C-M100-T60	1375	0.5	400	100
C-M80-T60	1375	0.5	400	100
C-M80-T180	1375	0.5	400	100
C-M80-T120	1375	0.5	400	100
C-M80-T30	1375	0.5	400	100
C-M60-T60	1375	0.5	400	100

3.3.2 Waste-Based Systems

Different mix design was also created in the present study to observe the pozzolanic activity. Theoretically, water, silica and/or aluminum-containing material and calcium hydroxide must be present in the matrix for the pozzolanic reaction to occur [91]. Considering this information, a mixture of pozzolanic material, calcium hydroxide, standard sand and water was designed. The design was inspired by the TS 25 standard [92]. Brick wastes which were grinded with different grinding parameters were used as pozzolanic material in the mixture design. This mixture design prepared for the compressive test analysis as shown in Table 3.10. Mix designs were prepared with brick waste which is grinded on different grinded parameters. Additionally, waste-based specimens were cured for 7 days in according to TS 25.

Table 3.10 Mixture content of waste-based systems

Specimen ID	Aggregate (g)	Water/Binder	Dry Mixture (g)		Ca (OH) ₂ (g)
			Cement	Mixed Brick Waste	
W-M120-T60	1350	0.5	-	295.86	150
W-M100-T60	1350	0.5	-	295.86	150
W-M80-T60	1350	0.5	-	295.86	150
W-M80-T180	1350	0.5	-	295.86	150
W-M80-T120	1350	0.5	-	295.86	150
W-M80-T30	1350	0.5	-	295.86	150
W-M60-T60	1350	0.5	-	295.86	150

3.4 Specimen Preparation and Curing

The cement and/or pozzolanic material content was named as dry mix. Firstly, the prepared dry mix ingredients were weighed and mixed by a laboratory mixer as shown in Figure 3.10 for one minute. Thus, the dry mixture was homogenized. Water was slowly added to turn the homogenized dry mixture into paste.



Figure 3.10 Mixer which is used to prepare mortar

After mixing for 1 minute at normal speed, standard sand was added and continued to mix at high speed for 30 seconds. Then the mixer was stopped, and the mortar mixture adhered to the bowl wall was cleaned with a gauging trowel within 30 seconds. The

mortar was taken back to the mixer and continued to mix at high speed for 1 minute. Thus, the mortar preparation procedure was completed.

Cement-based and waste-based mortar samples were placed in different molds to observe the pozzolanic activity. The waste-based mortar samples were placed in 40x40x160 mm metal molds, while the cement-based mortar samples were placed in 50x50x50 mm metal cube molds as shown in Figure 3.11.



Figure 3.11 40X40X160 mm and 50X50X50 mm metal molds respectively

A tamper was used as the equipment for the molding procedure. Firstly, 40x40x160 mm molds were half filled and each specimen was placed by tapping 32 times. Afterwards, the material was added to fill the molds completely and tapped 32 times again. The material remaining on the mold was removed by gauging trowel. The cement-based specimens were placed in the cube molds similarly. The samples placed in the molds were covered with plastic wrap to prevent water evaporation. The cube specimens were cured in oven at 24 °C for one day, then removed from the mold and stored under lime saturated water in oven at 24 °C for 7 and 28 days for testing. The specimens poured into 40x40x160 mm molds were cured in oven at 24 °C for the first day and then cured in oven at 55 °C for 6 days.

3.5 Experiment

Different test methodologies were used to observe the effect of grinding parameters on fineness of material, consistency, compressive strength and pozzolanic activity. In this context, PSD analysis was carried out to determine the average particle size of grinded materials. Flow table test was performed to observe the effect of grinding brick waste at different parameters on the consistency properties of the cementitious system. Waste-based and cement-based mix designs were used to observe the contribution of waste milled at different parameters on compressive strength. XRD, TGA, isothermal calorimetry and Frattini tests were performed to observe the effect of different grinding parameters on pozzolanic activity.

3.5.1 Flow Table Test

The ASTM 1437 procedure was followed for the determination of the flow properties [93]. Cementitious system of mixed grinded brick wastes with different grinding parameters was used as fresh mortar. Flow table test equipment as shown in Figure 3.12 was cleaned before use. The prepared fresh mortar mix was filled halfway into the mold. The fresh mortar mix was tapped with a temper 20 times to place the fresh mortar mix into the mold. Then, the remaining space was filled completely with material. The excess material remaining on the mold was cleaned with a trowel. One minute after placement, the mold was carefully removed. Then, immediately, the table was dropped 25 times within 15 seconds. The longest diameter of the material spread on the table and the diameter perpendicular to it were measured by ruler and the average value was noted.



Figure 3.12 Flow table apparatus

3.5.2 Compressive Strength Test

Compressive strength test was carried out separately for cement-based and waste-based mixture design as shown in Figure 3.13. Since the cement-based mixtures were poured into metal cube molds, the test procedure was carried out by following ASTM C109 [94]. For the cube specimens, the test was carried out at loading rate of 900 N/s. Waste-based mixtures were poured into 40x40x160 mm molds and the specimens were first split into two using a flexural testing machine. The compressive strength test of the split specimens was carried out at loading rate of 2400 N/s. Cube specimens were tested at 7 and 28 days. The beam specimens were tested on the 7th day. The compressive strength of 3 specimens was measured for each day and the average strength value was noted.

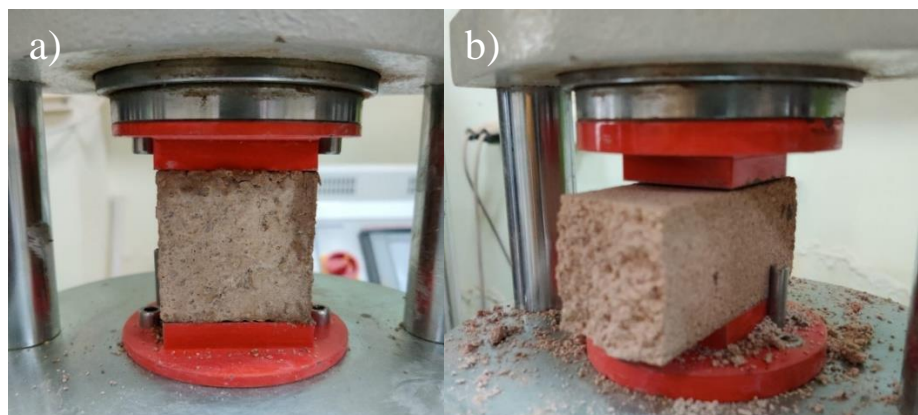


Figure 3.13 Compressive strength test demonstration, a) cube specimen, b) beam specimen

3.5.3 Particle Size Distribution

Powder materials are used in many different industries including construction industry. Different properties of the material such as mechanical strength and density are affected by the PSD characteristics of the material. Thus, a detailed assessment of powder material PSD is required since particle characteristics are significantly related to the quality of powder material. Due to its ability to successfully examine large numbers of particles, particle size analysis which is based on the concept of laser diffraction has been widely utilized in microparticle analysis. Laser particle size analysis based on the principle of light interaction with particles and this approach assumes that the particle shapes are spherical [95]. In this study Malvern Instruments Mastersizer 2000 as shown in Figure 3.14 is used as laser diffraction particle size analyzers to determine the PSD of the grinded brick wastes. All measurements were executed in dry dispersion of the instrument.



Figure 3.14 Malvern Instruments Mastersizer 2000 with dry dispersion unit

3.5.4 XRD Test

XRD is a nondestructive method that imparts detailed insights into a material's crystallographic structure, chemical composition. This technique relies on the constructive interactions of monochromatic X-rays with a crystalline specimen. X-rays which are characterized by shorter wavelengths, result from the declining of electrically charged particles possessing adequate energy. The generated X-rays are directed to a

sample in the XRD process. The interaction of these incoming rays with the sample generates diffracted rays. The diffracted rays are dispersed at various angles by the material. Dispersed rays are represented graphically to expose a unique distinctive diffraction pattern. Since materials have unique atomic arrangement, each material phase creates its own specific diffraction pattern [96]. Grinded brick waste and CH were mixed at a ratio of 2:1 by mass and formed a paste with 0.50 w/c ratio to investigate the effect of grinding parameters on pozzolanic activity. Prepared pastes were gently poured into 50 mL falcon tube and sealed to cure at 25 °C for 28 days as shown in Figure 3.15. Powder samples were taken from hardened pastes for XRD analysis. Rigaku Ultima-IV instrument was used for the XRD analysis and result were obtained at 2θ angle.



Figure 3.15 50 mL falcon tubes which is filled with paste

3.5.5 Thermogravimetric Analysis

TGA measures weight changes by heating the sample. Phase changes involve the release or absorption of heat due to the unique heat capacity of each material which is determined by its composition, internal vibrations, and structure [97]. TGA is considered as a direct method to investigate pozzolanic activity. Hydroxyl bonds start to break down after reaching a certain degree and this phenomenon occurs between 400 °C and 700 °C [98]. The powder samples were taken from pastes which have 2:1 weight ratio of brick waste and CH, with 0.50 w/c ratio. Pastes were cured at 25 °C for 28 days. Thermal analysis was performed on powder samples via TA Instruments (Q600 SDT) at a heating rate of 10 °C/min.

3.5.6 Isothermal Calorimetry Analysis

Isothermal calorimetry is a method for characterizing chemical processes that involves heat changes that occur spontaneously during the reaction. This technique measures the heat emitted or absorbed during the reaction [99]. Isothermal calorimetry test was performed on lime and brick waste pastes for 7 days by using a TAM Air Microcalorimeter (TA Instruments). A laboratory type mixer and deionized water were used to prepare the paste. The prepared samples were quickly placed in isothermal calorimetry sample holders. The thermal output of the paste was recorded in mW/g for 7 days. The total heat of reaction for the pastes was determined by integrating the thermal power versus time data.

3.5.7 Frattini Test

Frattini test was performed to test the potential pozzolanic activation of the grinded materials. Firstly, distilled water was boiled, and carbon dioxide was removed from it. Boiled distilled water was taken 100 milliliters with a pipette and transferred to a polyethylene container. The polyethylene container was kept in an oven at 40°C for 1 hour. Then, the prepared dry mixture (16 g CEM I and 4 g brick waste) was transferred to the polyethylene container with a funnel and shaken in the horizontal plane for 20 seconds before the container was sealed. The sample was kept in an oven at 40°C for testing after 8 and 15 days as shown in Figure 3.16. Separate containers should be prepared for each day of testing. Samples which do not give positive results within 8 days are tested again after 15 days. After 8 and 15 days, the containers were taken out from the oven and the solution in the container was filtered into a Buchner funnel within 30 seconds as shown in Figure 3.17. The filtration was carried out with filter paper with a dry average pore diameter of about 2 microns which was placed in the Buchner funnel. The filtered solution was isolated from the atmosphere to prevent contact with air. Filtered solution is maintained at the laboratory to cooldown to room temperature. 50 mL of the solution is transferred to a 250 mL flask with a pipette to calculate the hydroxyl ion concentration. Afterwards, 5 drops of methyl orange indicator are dripped into the solution in the flask and titrated with hydrochloric acid by using burette. The turning point of the titration is understood when the color of the solution turns into orange yellow. After the solution turned into orange yellow, the volume of consumed hydroxyl acid solution

was noted and used in the following equation to calculate the concentration of hydroxyl ion; $[\text{OH}]^- = 2 \times V \times f$ where V is volume of hydrochloric acid solution consumed in the titration process in terms of mL, f is factor of hydrochloric acid solution in terms of g/mL and $[\text{OH}]^-$ is hydroxyl concentration in terms of mmol/L.



Figure 3.16 Samples in the oven for curing

The remaining titrated solution was used for the determination of calcium oxide concentration. Firstly, the pH value of the remaining solution was set to 12.5 ± 2 by dripping sodium hydroxide solution. A pH meter was used for this process. Then 0.1 g of calcon indicator was added to the solution and the titration was performed with ethylenediaminetetraacetic acid (EDTA) solution. Similarly, EDTA solution was added drip by drip into the solution in the flask by using a burette. The turning point was understood when the color of the solution changed from pink to blue. The volume of EDTA solution consumed on the burette was noted and used in the following equation for the determination of calcium oxide concentration; $[\text{CaO}] = 0.6 \times V \times f$ where V is volume of EDTA solution consumed in the titration process in terms of mL, f is factor of EDTA solution in terms of g/mL, $[\text{CaO}]$ is calcium oxide concentration in terms of mmol/L.



Figure 3.17 Vacuuming the solution to filter

Calcium oxide and hydroxyl concentrations obtained from the equations were used to evaluate the pozzolanic property. The saturated concentration of calcium ion (expressed as calcium oxide) at 40°C was plotted as a function of hydroxyl ion to evaluate the pozzolanic property. The function curve was given as $[CaO] = 350 / ([OH] - 15)$ which represents maximum CaO concentration of the sample in solution. The values of hydroxyl and calcium oxide concentrations in the solution were placed on the graph and the samples under the graph successfully passed the experiment. The decrease in concentration was calculated mathematically. Calculations and experimental procedures were performed separately for each sample [100].

4. RESULTS AND DISCUSSION

4.1 Investigation of MFR Effect on Brick Waste

In this section, grinded brick waste which is grinded at different MFR values will be investigated. MFR effect on brick waste is investigated by particle size, fresh and hardened mortar performance and finally pozzolanic activity at constant grinding duration (60 minutes). 60, 80, 100 and 120% MFR values are investigated in this section.

4.1.1 Effect of MFR on Particle Size of Brick Waste

The microstructural characteristics of brick waste grinded at different amounts in a ball mill were investigated. In this study, the grinding duration was kept constant at 60 minutes to observe the effect of MFR. The particulate distribution of brick waste grinded at different MFR percentages (60%, 80%, 100% and 120%) which is shown in Figure 4.1.

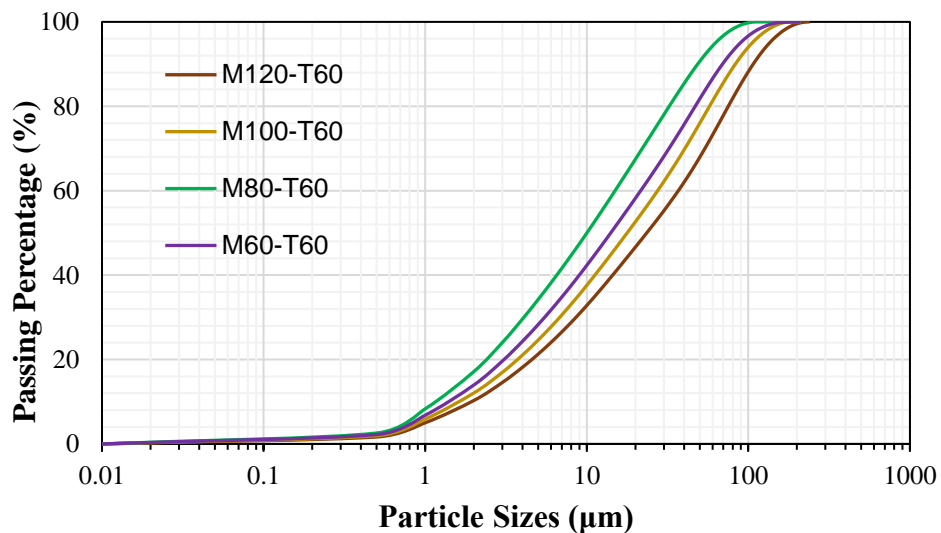


Figure 4.1 Particle size distribution of grinded brick wastes at different MFR

The D (0.5) value of the sample with 60% MFR was 14.07 µm, while the D (0.5) value was 10.02 µm for 80% MFR, 17.78 µm for 100% MFR and 23.51 µm for 120% MFR as shown in Table 4.1. According to the data, decrease in particle size was observed when the MFR percentage was increased from 60% to 80%. However, increase in particle size was observed when the MFR percentage was changed to 100% and 120%. This is an

indication that the grinding performance declines as the MFR percentage increases after 80%. Lower article size with 80% MFR can be explained because of the grinding particles in the chamber rapidly hitting each other or the chamber wall [101,102]. Declining in milling efficiency with the rising material feeding observed in the results after % 80 MFR. The increased powder feeding may result in an elevated viscosity of the material. It is stated that viscosity exhibits a linear relationship with material feeding until a critical point. In addition, the augmented viscosity caused by increased material feeding will consequently lower the collision velocity of the grinding media in the material due to the amplified drag force. Thus, higher average particle size in higher MFR values (% 100 and % 120) were observed [103]. It is seen that the optimum particle size is obtained at 80% MFR to achieve the lowest average particle size for 60 minutes grinding.

Table 4.1 D (0.5) values of brick wastes at different MFR

Specimen ID	D (0.5)
M120-T60	23.51 μm
M100-T60	17.78 μm
M80-T60	10.02 μm
M60-T60	14.07 μm

4.1.2 Effect of MFR on Fresh and Hardened Mortar

The flow table test results were utilized to evaluate the effects of MFR values on the fresh properties of cementitious mixtures containing ground brick waste, as shown in Figure 4.2. The results revealed that regardless of the grinding parameters, brick waste reduces the workability, and the reduction varies up to 21.7%. This can be explained by several factors. Firstly, the rough surfaces and angular shapes of the brick waste increase internal friction. Secondly, the irregular microstructure and porous structure of the clay-based brick waste demands higher water [104–107]. Furthermore, the results showed that there were variations in the workability performance of the mortar depending on MFR. It was observed that the workability increased with lower particle size. This can be attributed to the decreasing particle size, which reduces the presence of pores of the particles. Therefore, lower particles reduce the water demand [107]. In addition, as the size of the

particles decreases, their microstructure becomes more regular. Thus, the friction within the matrix decreases [104,108].

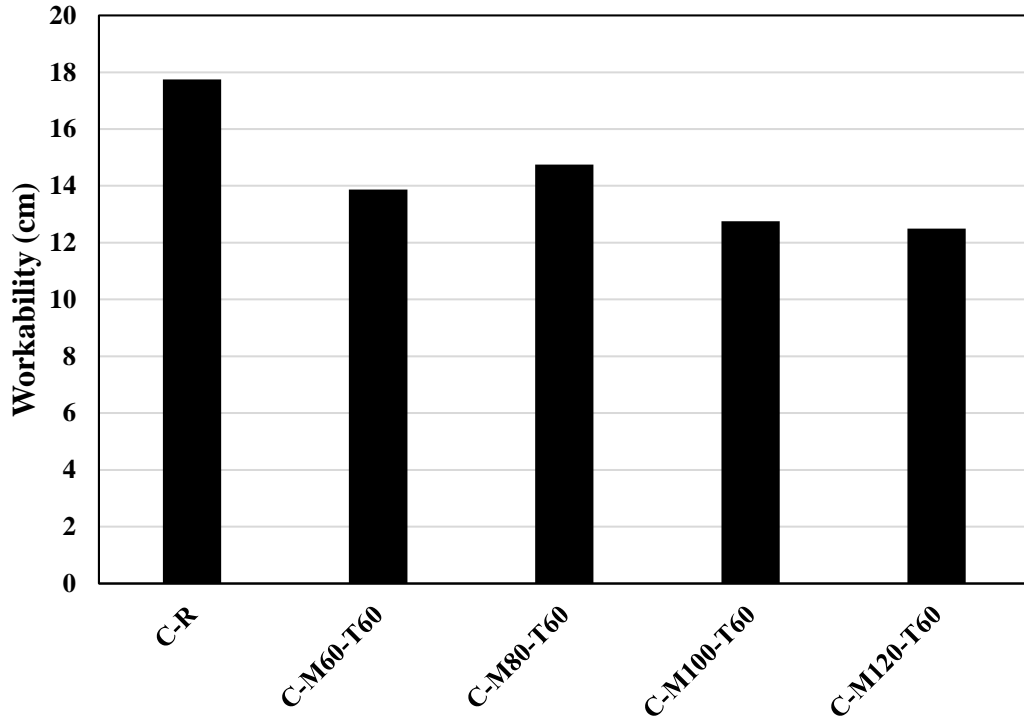


Figure 4.2 Workability results of cementitious mortar with brick waste substitution at different MFR

Figure 4.3 shows the effect of MFR value on compressive strength. Considering the 7-day strength results, the reference mix has the highest value of 38.7 MPa. It is obvious that the strengths of the materials grinded with MFR values of 100% and 120% have lower strengths than the material grinded with 80% MFR. 33.8 MPa for 60% MFR, 35 MPa for 80% MFR, 33.6 MPa for 100% and 31 MPa for 120% MFR results were obtained as shown in Figure 4.3. When the 28-day strength results are analyzed, it is seen that similar correlations occurs. In addition, the strength result of 80% MFR is higher than 120%, 100% and 60% MFR values. 49 MPa for reference mix, 46.1 MPa for 60% MFR, 46.9 MPa for 80% MFR, 42.4 MPa for 100% MFR and 40.3 MPa for 120% MFR results were received. To sum up, grinding brick waste with 80% MFR parameter reaches 90% and 97% strength activity index at 7-day and 28-day compressive strength results respectively.

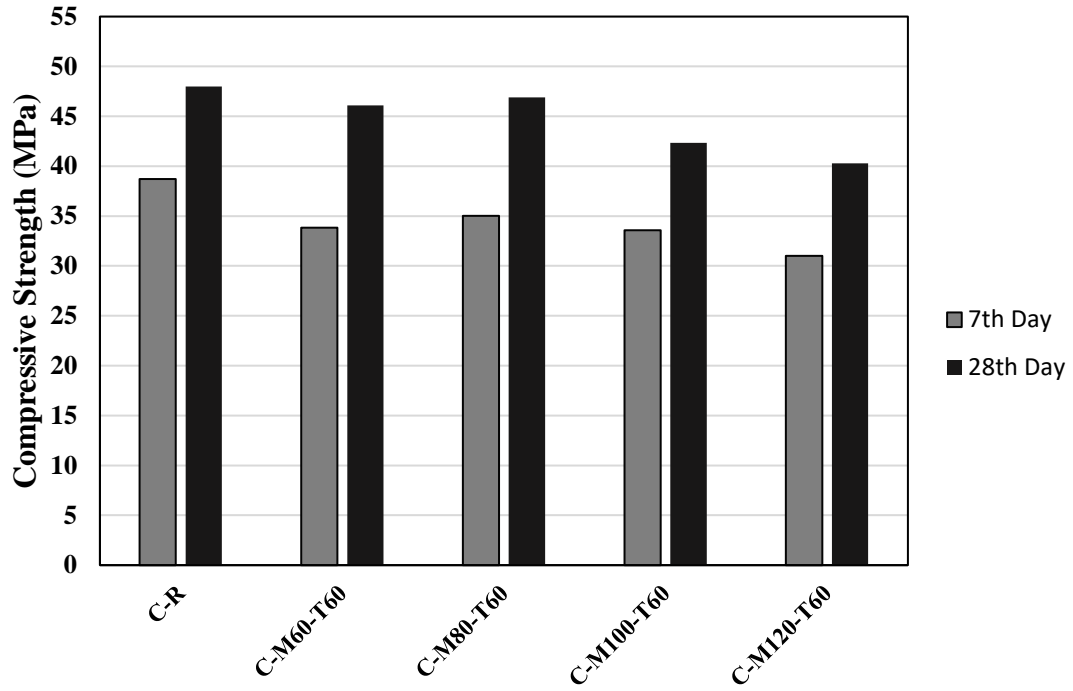


Figure 4.3 Compressive strength of cementitious mortars with brick waste substitution at different MFR

The decrease in early strength compared to the reference can be attributed to insufficient hydration of brick waste in the matrix [104,109]. However, the strength reduction caused by the grinded brick substitution was eliminated by the modifications of the grinding process. The increase in strength with grinded brick substitution under different conditions indicates pozzolanic reactivity. Since the brick waste includes SiO_2 and Al_2O_3 oxide composition, pozzolanic reaction occurs and more C-S-H gels form [105,109]. The finer brick waste reduces the size and number of pores, which leads to a stronger and denser microstructure [53,72,107].

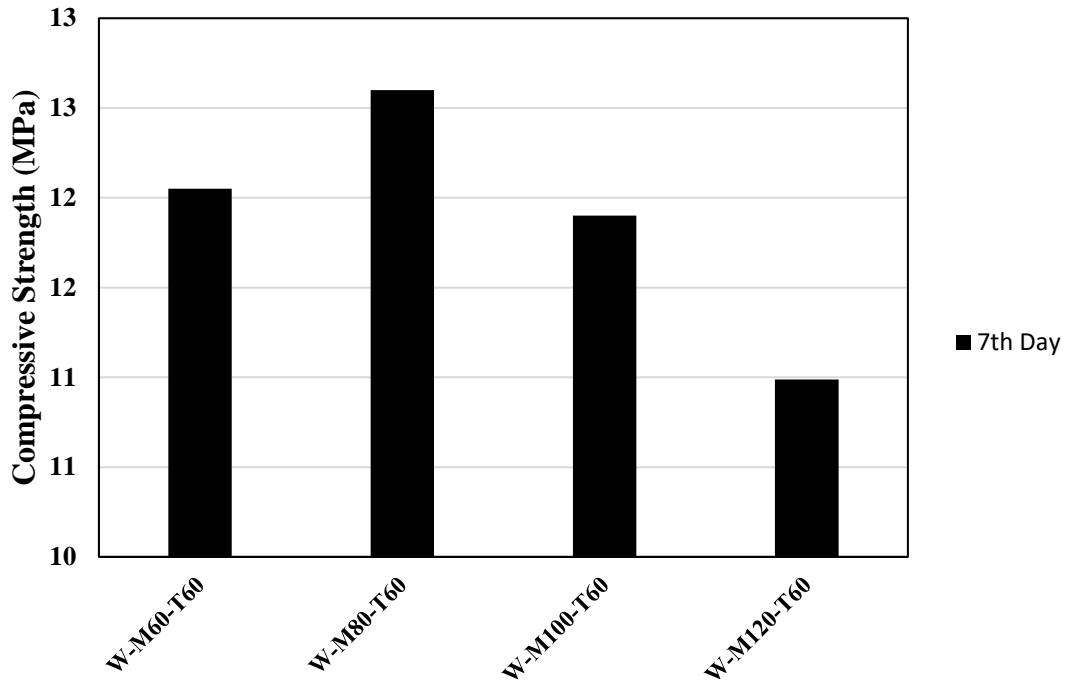


Figure 4.4 Compressive strength of waste-based mortars at different MFR

In waste-based castings, brick waste was used as binder and CH as activator. Results were obtained after 7 days curing in according to TS 25. The contribution of grinding parameters to the strength is shown in Figure 4.4. There is about 1 MPa difference between 60% MFR and 80% MFR. However, when the value reaches 100% and 120% MFR, the strength decreases to 11.9 MPa and 11 MPa, respectively. Considering the values, the reaction of brick waste with CH increased as the particle size decreased. This can be correlated with the grinding efficiency. Results show that increasing MFR value positively effects the grinding process up to 80%. However, further MFR values negatively effects grinding process. It can be said that, grinding process at 80% MFR increases surface of the waste to improve interaction between particles and CH. Thus, additional C-S-H gels were formed [105,109]. In this test result series, 80% MFR was found to be optimum.

4.1.3 Effect of MFR on Pozzolanic Activity

Different test methodologies were utilized such as XRD, TGA, isothermal calorimetry and Frattini to investigate the MFR effect on pozzolanic activity of brick waste. CH consumption and cumulative heat release are considered to determine pozzolanic activity.

4.1.3.1 XRD Analysis of Waste-Based Paste

XRD analysis was performed on the prepared pastes to investigate the pozzolanic reaction of brick wastes which were grinded at different MFR values. The test evaluated the CH diffraction peaks in the paste. CH diffraction peaks are visible at 18.15° , 34.25° and 47.28° . The highest CH diffraction peak was observed in the paste prepared with M120-T60 as seen in Figure 4.5. The reduction of the diffraction peaks reveals that the grinding process generally increases the CH consumption. The decreasing CH diffraction peak is related to the increasing degree of reaction of the grinded material [110].

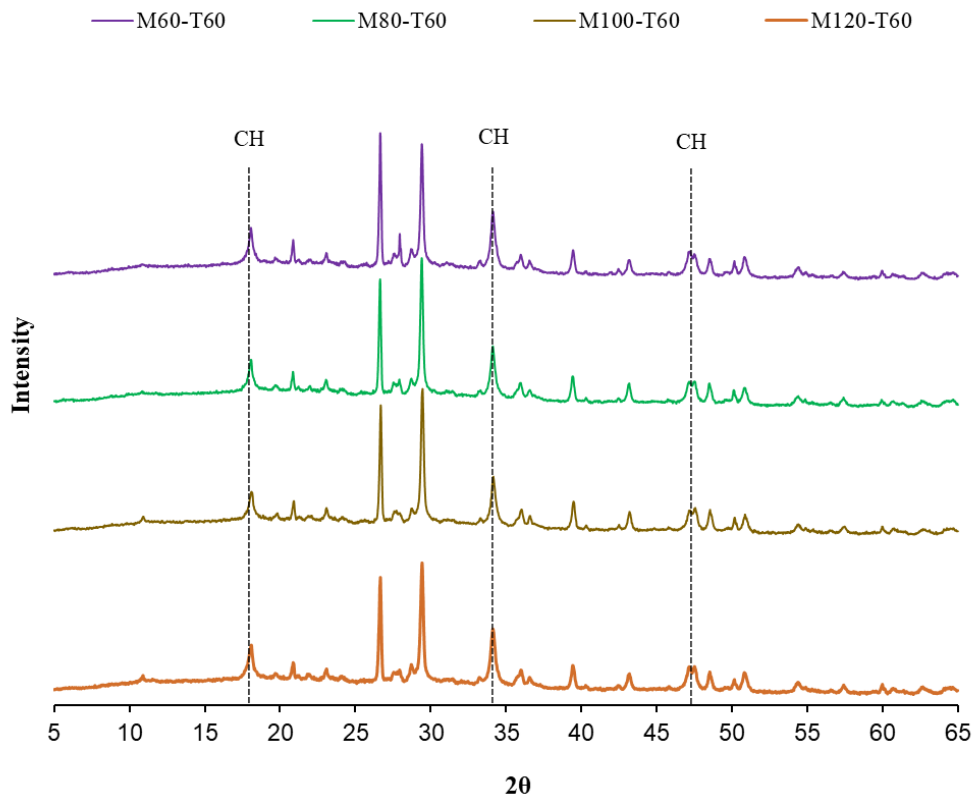


Figure 4.5 MFR effect on XRD pattern of waste-based paste

4.1.3.2 TGA Analysis of Waste-Based Paste

TGA test executed on brick waste pastes at different MFR. It is stated that the temperature range of dihydroxylation of CH is 375-450°C [111]. Therefore, the weight losses of the waste-based pastes were calculated between 375°C and 450°C. At 60% MFR, the dihydroxylation loss in the ground sample is 2.6%, while the dihydroxylation loss is 2.51% at 80% MFR as seen in Figure 4.6. This shows that the amount of CH in the matrix decreases with an MFR of 80%. However, as the MFR value increases to 100% and 120%, dihydroxylation weight loss increases. This situation shows that lower particle size increases the CH consumption of the system. Decreasing CH amount is related to the contribution of CH into hydration products [112]. This indicates that 80% MFR has more pozzolanic reaction degree [113].

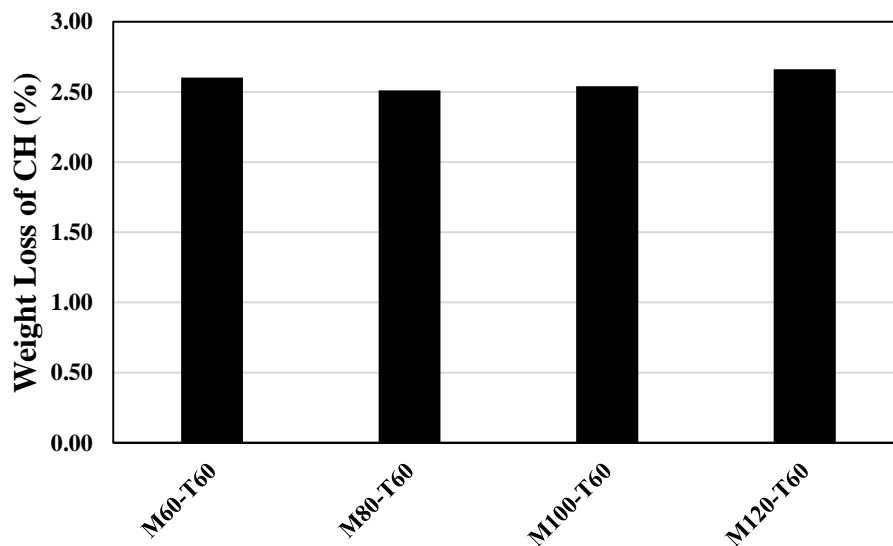


Figure 4.6 Dihydroxylation weight loss of brick wastes at different MFR

4.1.3.3 Isothermal Calorimetry Analysis of Waste-Based Paste

The cumulated heat release of isothermal calorimetry results is shown in Figure 4.7. Cumulated heat release is directly related to pozzolanic activity and the degree of reaction with supplementary cementitious materials (SCM) [114]. Hence, higher values of cumulated heat release indicate increased pozzolanic reactivity of SCM [98,115]. The results showed that increasing the MFR up to 80% improves the activity of waste brick and enhances the cumulative heat release. However, increasing the MFR above 80%

reduced the cumulative heat release. In addition, the cumulated heat release reached the lowest level when the MFR was 120%. This phenomenon may be related to particle size which is affected by grinding efficiency. Grinding efficiency declines as the MFR percentage increases after 80%. This can be explained by lower ball impact and movement in the chamber. Higher material feed ratio prevents the grinding media to work efficiently [101,102]. Results show that waste activity increases by effective grinding at 80% MFR parameter.

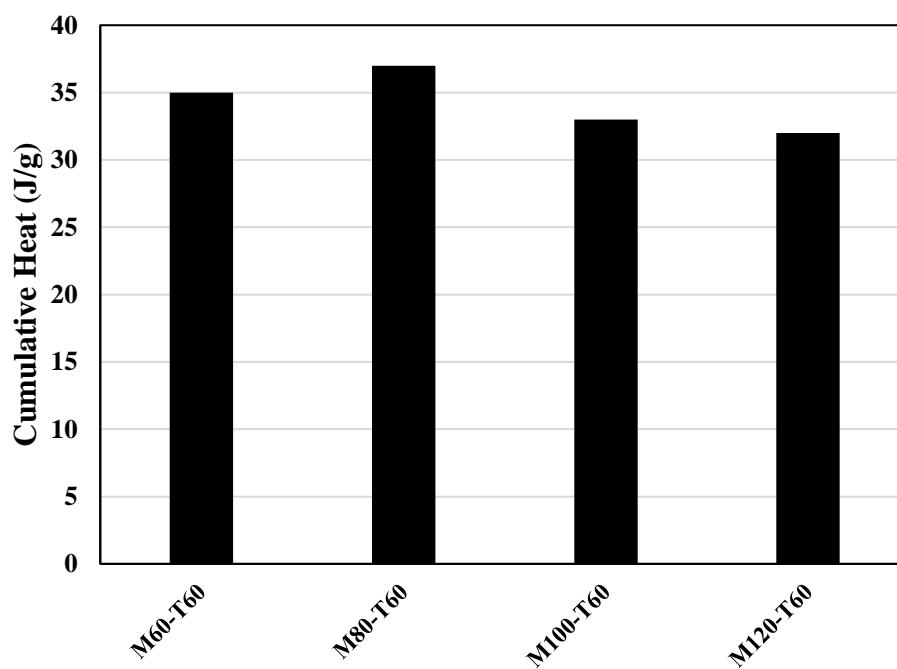


Figure 4.7 Cumulative heat evolution of brick wastes at different MFR

4.1.3.4 Frattini Test Analysis of Brick Waste

Frattini test results were above the solubility curve on the 8th day and the test was extended to the 15th day. Results were placed on the graph and compared with CH solubility curve as described in BS EN 196-5. According to the data obtained, it was observed that the brick waste grinded at 80% MFR was below the curve. The other MFR ratios (60%, 100% and 120%) did not satisfy the test. Only 80% MFR satisfied the pozzolanic activity test. 80% MFR contributed to the formation of C-S-H gel by reducing the calcium ion concentration in the system. [116]. Therefore, 80% MFR exhibited the optimum degree of pozzolanic reaction. The results show that the brick waste was grinded to finer particle

size with the MFR value of 80% and there is a correlation between particle size and pozzolanic reaction. Since brick waste has quartz-based material and inert, 60%, 100% and 120% MFR parameters at 60 minutes grinding duration is not sufficient to satisfy the pozzolanic activity test. In Figure 4.8, 80% MFR is very close to the solubility curve, thus it barely satisfies the test. Increasing grinding duration is required for further investigations.

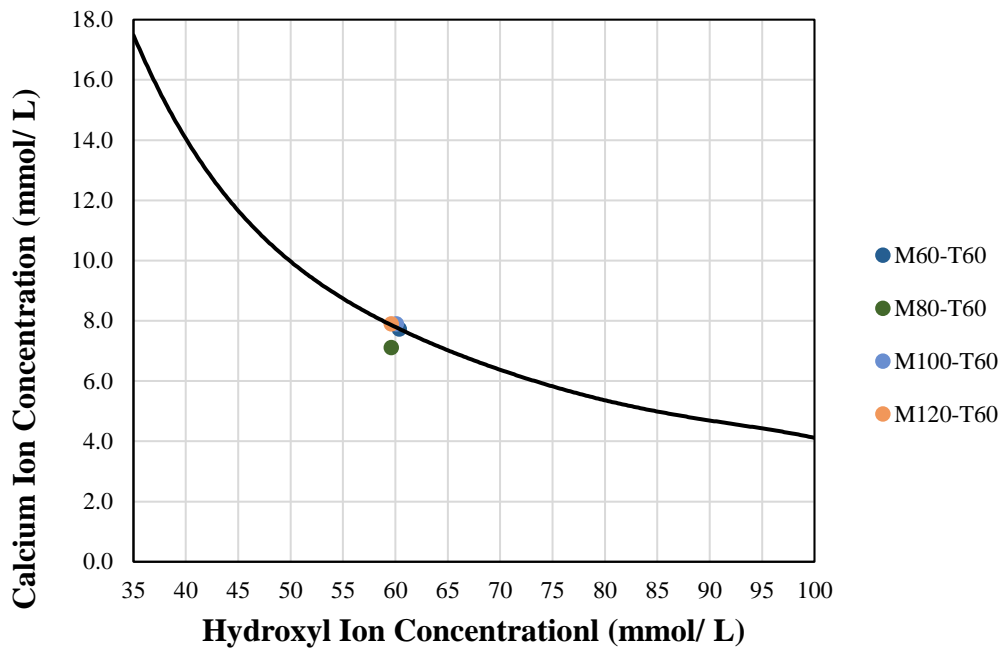


Figure 4.8 Frattini results of brick wastes at different MFR

4.2 Investigation of Grinding Duration Effect on Brick Waste

In this section, grinded brick waste which is grinded at different grinding durations will be investigated. Different grinding duration effect on brick waste is investigated by particle size, fresh and hardened mortar performance and finally pozzolanic activity at constant MFR (80%).

4.2.1 Effect of Grinding Duration on Particle Size of Brick Waste

80 % MFR was selected as the optimum value based on the previous experimental results. The MFR was maintained constant at 80% and the brick waste was grinded in different

durations (30, 60, 120 and 180 minutes) to observe the effect of grinding time. Table 4.2 shows that decreasing the grinding duration to 30 minutes increases the particle size. In addition, particle size decreases with increasing grinding time.

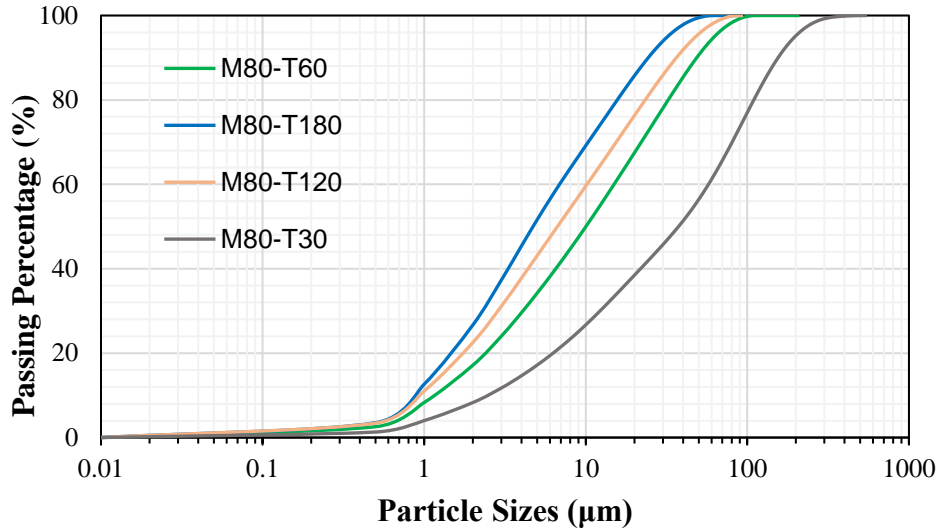


Figure 4.9 Particle size distribution of grinded brick wastes at different grinding duration

While $D(0.5)$ value was $6.68\mu\text{m}$ in 120 minutes, $D(0.5)$ value was $4.72\mu\text{m}$ in 180 minutes grinding as shown in Table 4.2. Energy transfer duration between the grinding median and the material decreases in shorter grinding time. Moreover, it can be said that higher the grinding duration increases the collision duration of the material with grinding median and chamber wall [101,102]. Thus, a decrease in particle size was observed by increasing the grinding duration as shown in Figure 4.9. Results showed that the smallest particle size was obtained by 180 minutes grinding. It should be noted that prolonged grinding duration increases the energy consumption.

Table 4.2 $D(0.5)$ values of brick wastes at different grinding duration

Specimen ID	$D(0.5)$
M80-T30	$37.51\ \mu\text{m}$
M80-T60	$10.02\ \mu\text{m}$
M80-120	$6.68\ \mu\text{m}$
M80-T180	$4.72\ \mu\text{m}$

4.2.2 Effect of Grinding Duration on Fresh and Hardened Mortar

Figure 4.10 shows that the workability value increases as the grinding duration increases. The workability value obtained at the longest grinding duration (15.5 cm) is the closest to the reference value.

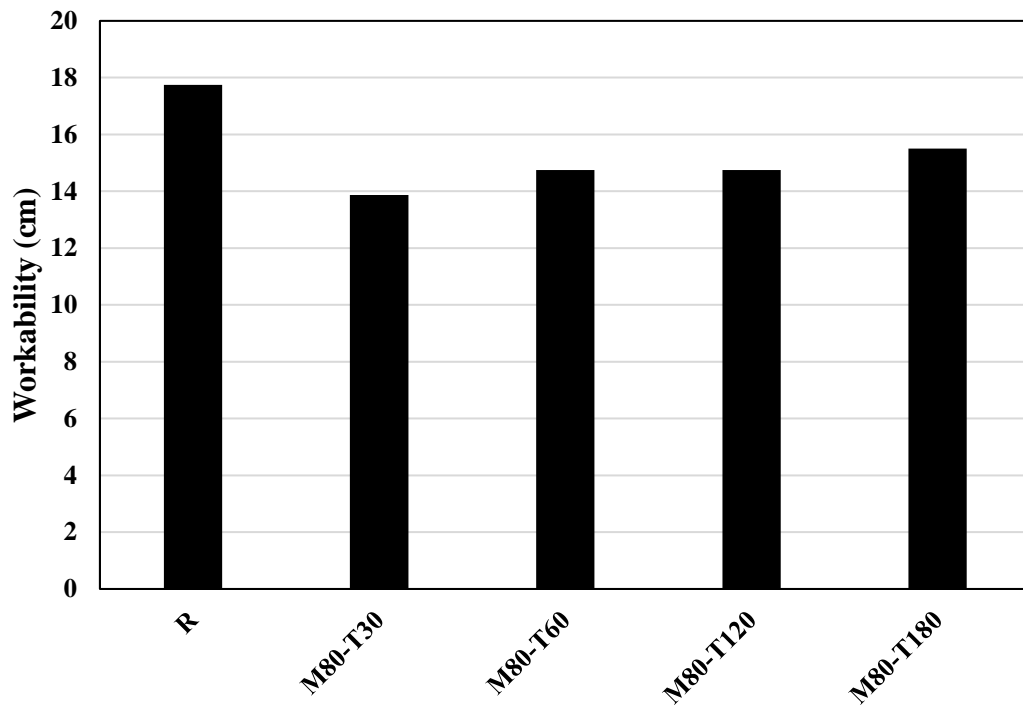


Figure 4.10 Workability results of cementitious mortar with brick waste substitution at different grinding duration

Decreasing particle size by increasing grinding duration enhances the workability value by 11.5%. The water demand increased compared to the reference sample, even though the workability values improved by grinding duration. In general, the increase in water demand is due to the porous structure of clay-based brick waste [104–107]. The material, which was grinded for longer duration, became finer. Thus, its porous structure decreased, and particles became more rounded shape. This phenomenon decreased the matrix viscosity [71,104,107,108]. As a result, the water demand decreases for the waste brick which is grinded for longer duration.

Effects of grinding duration on compressive strength are shown in Figure 4.11. Results show that increasing grinding durations positively affects compressive strength in early age. In addition, 180 minutes grinding duration increased the compressive strength by 16.9% with respect to 30 minutes grinding.

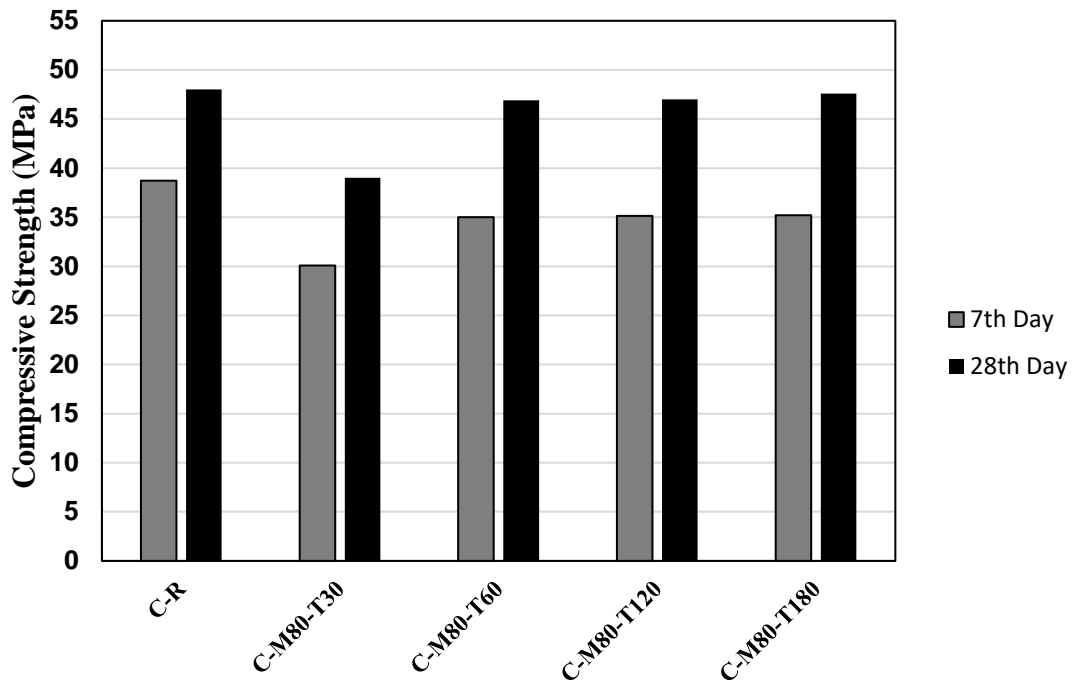


Figure 4.11 Compressive strength of cementitious mortars with brick waste substitution at different grinding duration

Figure 4.11 shows that the grinding time has no effect on the early strength after 60 minutes. This may be due to the insufficient reaction of the brick waste [104,109]. When the late strength values are analyzed, the positive effect of grinding duration on strength is apparent. The late strength of the reference sample reached 48 MPa. When the brick waste is ground for 180 minutes, the late strength is 47.6 MPa. Thus, the compressive strength gets closer to the reference value in late strength. The decreasing particle size due to the increase in grinding duration enhanced the degree of pozzolanic reaction and created a filler effect in the matrix. Thus, extra C-S-H gels were formed, and the strength increased [104,105,109].

The contribution of grinding durations to the strength in according to TS 25 is shown in Figure 4.12. Higher grinding duration positively affected the compressive strength results. Compressive strength of brick waste grinded for 30 minutes and 60 minutes was 9.1 MPa and 12.6 MPa, respectively. Increasing the grinding duration up to 180 minutes enhanced the compressive strength result to 13.5 MPa. The increased strength values due to the higher grinding duration can be attributed to the reaction between CH and the material. Decreasing particle size increased the degree of pozzolanic reaction and formed C-S-H gel [105,109].

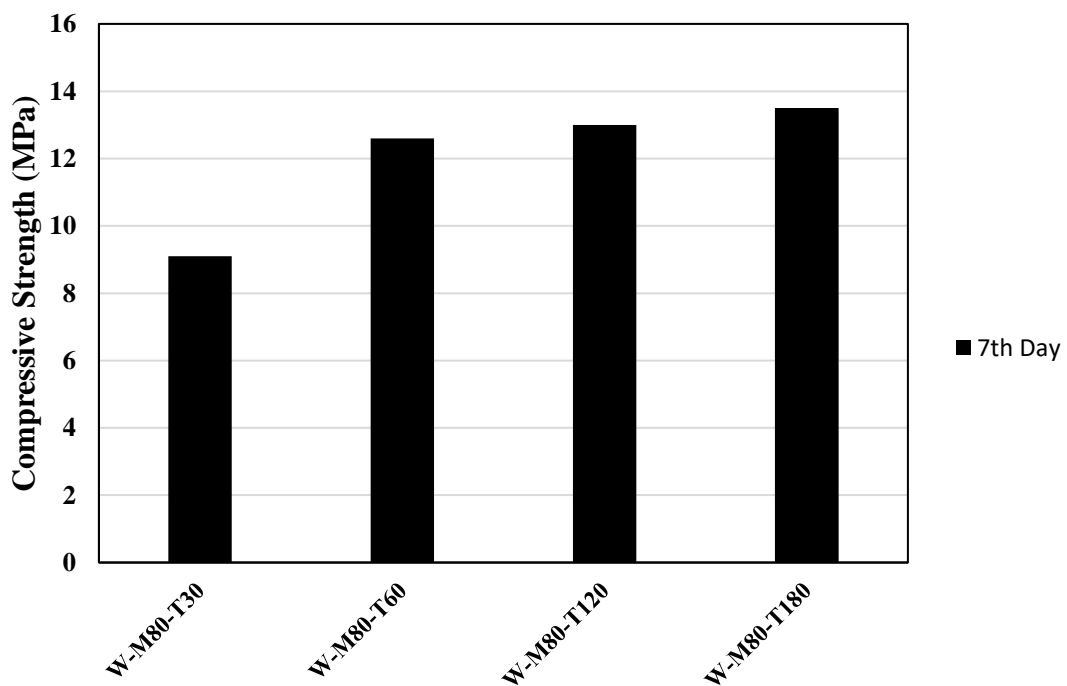


Figure 4.12 Compressive strength of waste-based mortars at different grinding duration

4.2.3 Effect of Grinding Duration on Pozzolanic Activity

Different test methodologies were utilized such as XRD, TGA, isothermal calorimetry and Fratini to investigate the grinding duration effect on pozzolanic activity of brick waste.

4.2.3.1 XRD Analysis of Waste-Based Paste

XRD analysis was performed on the prepared pastes to investigate the pozzolanic reaction of brick wastes which were grinded at different grinding durations. M80-T30 has considerably higher CH diffraction peak at 34.25 as seen in Figure 4.13. The test results show that CH diffraction peaks decline by increasing grinding duration. This might indicate CH was consumed by the material to initiate pozzolanic reaction [20,110].

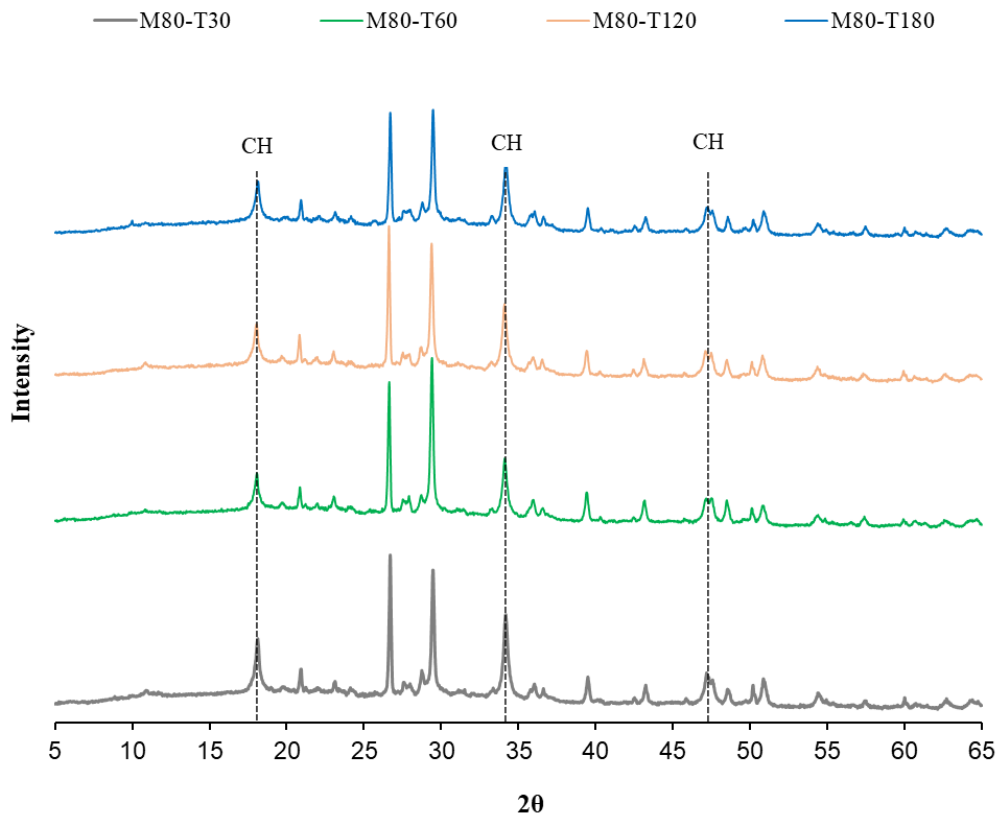


Figure 4.13 Grinding duration effect on XRD pattern of mixed brick waste paste

4.2.3.2 TGA Analysis of Waste-Based Paste

TGA results of brick waste at different grinding duration are shown in Figure 4.14. Firstly, results show that 30 minutes grinding duration decreases the weight loss of CH. Secondly, increasing grinding duration to 60 minutes decreased dihydroxylation weight loss by %9 while 120 minutes grinding duration reduced weight loss of dihydroxylation to 2.29%. Finally, 180 minutes of grinding duration caused reduction on weight loss of dihydroxylation by 26.1% with respect to 30 minutes of grinding. It is obvious that

grinding duration directly effects the CH consumption in the matrix. Additionally, it can be stated that exceeding CH weight didn't contribute the pozzolanic reaction. However, increasing grading duration decreases the particle size and enhances pozzolanic reaction degree [113].

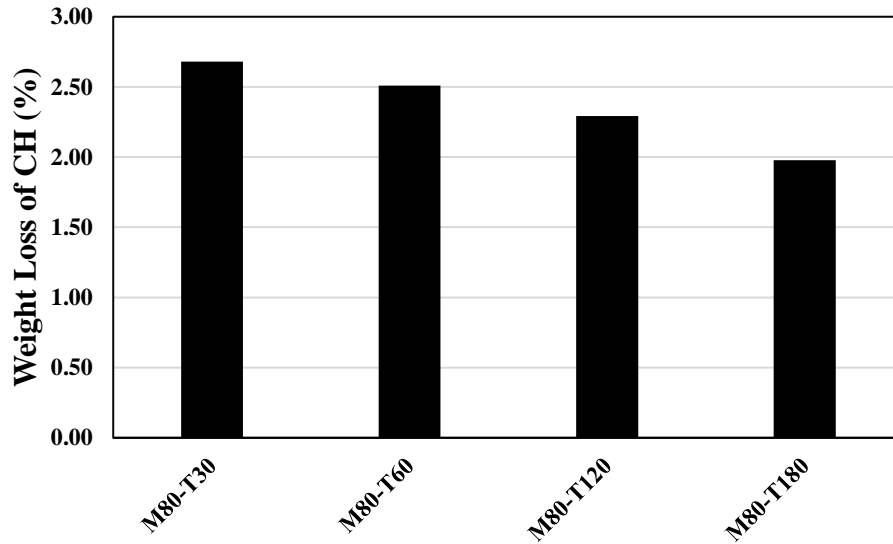


Figure 4.14 Dihydroxylation weight loss of brick wastes at different grinding duration

4.2.3.3 Isothermal Calorimetry Analysis of Waste-Based Paste

The accumulated heat release of isothermal calorimetry results is shown in Figure 4.15. 30 minutes grinded brick waste present the lowest cumulative heat release among the others. Increasing grinding duration to 180 minutes enhanced the cumulative heat release by %40. 180 minutes grinding duration provides lower particle size and higher heat cumulative heat release as seen in Figure 4.15. Therefore, there is a correlation between particle size and cumulative heat release. Heat release indicates pozzolanic reactivity. Thus, higher heat release is related to higher pozzolanic reaction degree [98,114,115]. Test results show that increasing grinding duration increase the degree of pozzolanic reaction.

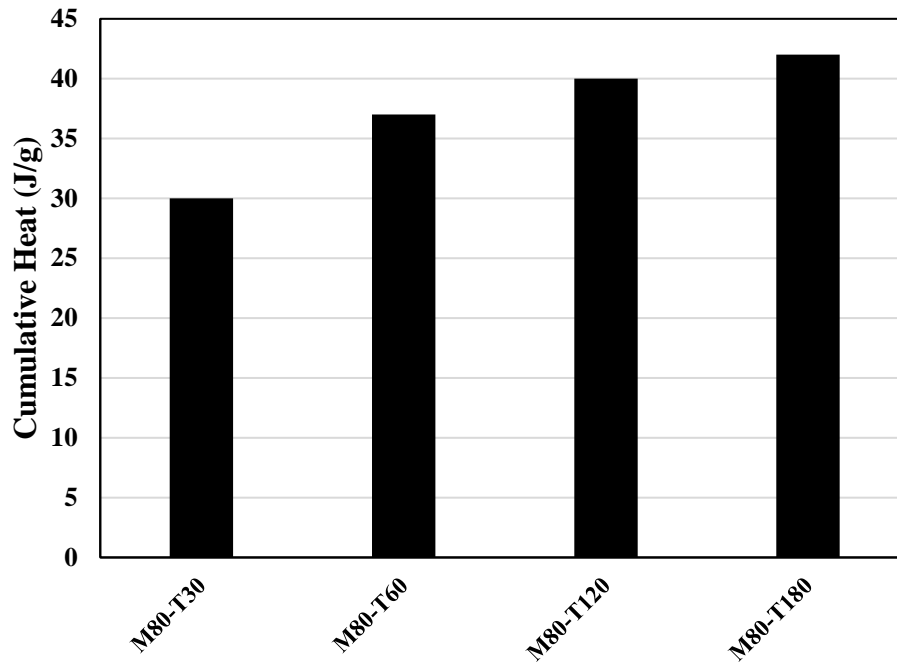


Figure 4.15 Cumulative heat evolution of brick wastes at different grinding duration

4.2.3.4 Frattini Test Analysis of Brick Waste

Frattini test results did not satisfy the pozzolanic activity according to BS EN 196-5 on 8th day. Therefore, test was extended to the 15th day. 30 minutes grinding duration did not satisfy the test as seen in Figure 4.16. However, increasing grinding duration influenced the test results positively. 60, 120 and 180 minutes of grinding duration enhance the pozzolanic activity of the brick waste and satisfy the test. Additionally, results show that higher grinding duration increases calcium ion consumption in the matrix. 60 minutes grinding provides %9.43 CH reduction while 120 minutes and 180 minutes grinding present %9.57, %17.82 respectively. As seen in Figure 4.16, calcium ion concentration is decreasing while the grinding duration is increasing. This phenomenon directly related to the degree of pozzolanic reaction. Thus, higher grinding durations with %80 MFR react with CH in the matrix and form C-S-H gels [116]. In general, results show that grinding duration improves pozzolanic properties of the brick waste. This can be related to the particle size of the material.

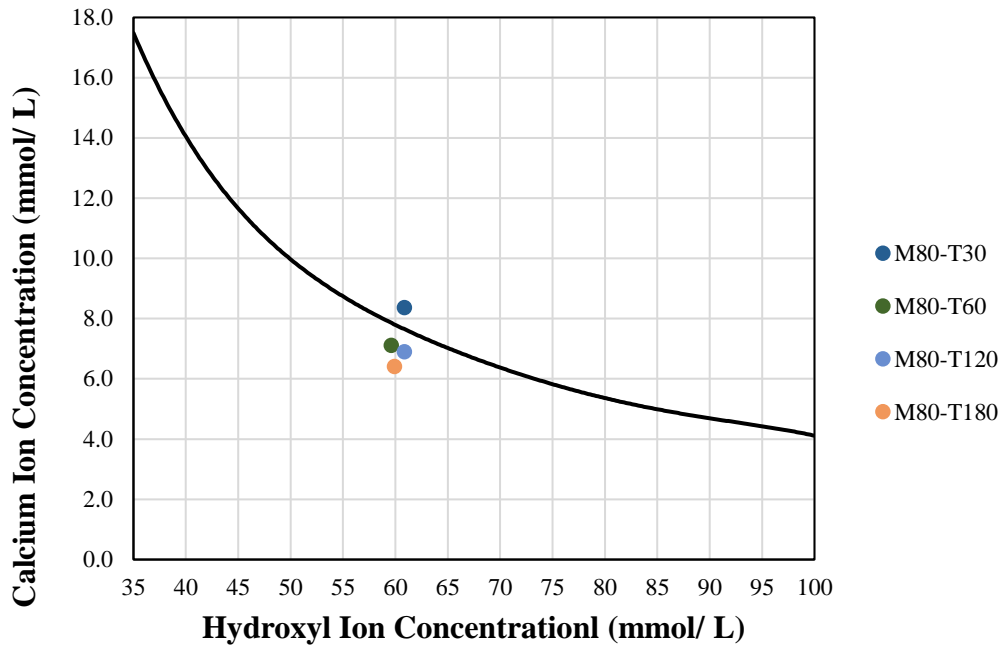


Figure 4.16 Frattini results of brick wastes at different grinding duration

Since compressive strength test methodology cannot distinguish between pozzolanic activity and filler effect, pozzolanic activity test should be supported by another test methodology. Results show that Frattini test provides accurate results to determine the pozzolanic activity of the waste material. In addition, TGA, XRD analysis and isothermal calorimetry analysis are effective to observe CH consumption and reaction degree. However, these methods do not put limit value to determine if the material is pozzolanic. Thus, chemical methods such as Frattini test might be considered to investigate pozzolanic activity.

To conclude, grinding process enhances pozzolanic activity of the material dramatically. In addition, optimum grinding parameters are necessary to achieve mechanically activated material. In this study, it is obvious that 80% MFR and 60, 120 and 180 minutes of grinding process satisfy pozzolanic activity test. However, increasing grinding duration negatively effects energy consumption. For further investigations, grinding aids can be used to decrease the grinding energy consumption while ensuring the desired pozzolanic properties of the material.

5. CONCLUSIONS

In this thesis, different types of brick waste were collected from different construction sites to demonstrate average properties of the bricks. RT, HB and RCB are collected as brick wastes. All the wastes transformed into laboratory and classified. All wastes crushed into smaller parts to conduct test methodology. All crushed brick wasted then, mixed in the same proportion to represent average waste chemical and mechanical properties. Mechanical and chemical properties of mixed brick wastes were characterized by utilizing pycnometer, XRF and XRD. Different grinding parameters were selected to observe effect of grinding parameters on pozzolanic reaction of mixed brick wastes. The comprehensive methodology procedure is executed, which is including TGA, XRD, isothermal calorimetry, Frattini, and compressive strength tests. Thus, understanding of the pozzolanic activity under different grinding conditions is provided about brick waste. In addition, grinding parameters were examined which effected pozzolanic activity.

Conclusions which are obtained from test results are arranged as following:

- Firstly, grinding effect of various MFR is investigated. MFR values vary between 60%, 80%, 100% and 120% at constant grinding duration and BFR. The smallest D (50) value was obtained at 80% of MFR. In addition, D (50) values increased up to 80% and then started to decrease. Therefore, 80% of MFR is the optimum operation process for grinding in this study. Secondly, effect of grinding duration is studied. Different grinding durations were determined as 30, 60, 120 and 180 minutes at constant MFR of 80% and BFR which is %20. Results show that there is gradually increase in D (50) values by the grinding duration increases. Thus, 180 minutes of grinding duration yields smaller D (50) value among the other grinding parameters. It can be related to the higher energy transfer between grinding median and material. Additionally, collapsing between particles and chamber wall increases energy transfer which enhance the grinding process.
- Compressive strength results show that strength gain is corelated with particle size of the brick waste. However, early strengths could not reach the reference strength

which is 48 MPa. Results demonstrate that late strength reaches to reference strength value by decreasing D (50) value. Thus, the highest late strength is obtained at 80% of MFR and 180 minutes grinding duration which is 47.6 MPa. Additionally, waste-based compressive strength results also support this argument. 180 minutes grinding duration at 80% MFR yields the highest compressive strength value which is 13.5 MPa. It can be related to the pozzolanic reaction, lower particle size enhances the pozzolanic reaction degree. Therefore, higher compressive strength is obtained at later age.

- Workability test results demonstrate that increase in consistency has a correlation between particle size. In addition, lower particle size enhances consistency property of the brick waste. Thus, the best consistency value is obtained at 80% of MFR and 180 minutes grinding duration among the other grinding parameters. Even though the highest consistency value is obtained at specific grinding parameters, workability is lower than reference mix by 12.7%.
- XRD results show that grinding the brick waste directly effects the CH consumption. Lower CH diffraction peaks are occurring on the grinded material. In addition, TGA test results confirm that 80% of MFR at 180 minutes grinding duration performs more CH consumption among other grinding parameters. TGA test results show that grinding the brick waste 180 minutes increases the dihydroxylation by 28%. Since CH consumption related to the pozzolanic reaction degree, it is obvious that grinding process enhances the pozzolanic activity of the waste material.
- Frattini and isothermal calorimetry test results have a correlation between TGA results. Isothermal calorimetry test results show that optimum MFR value is 80% with higher cumulative heat value which is 37 J/g. In addition, grinding duration increases cumulative heat. By grinding brick waste 180 minutes increases the cumulative heat by %13.5 with respect to the 60 minutes grinding at 80% MFR. Frattini results show that 80% MFR value at 60 minutes grinding duration meets the requirement of the pozzolanic activity according to BS EN 196-5. Longer grinding duration directly effects the CH consumption. Test results of Frattini test show that 180 minutes of grinding increases the CH consumption by %89 with

respect to the 60 minutes of grinding. In addition, Frattini test results have direct correlation with compressive strength results. Material with higher CH consumption yields higher compressive strength.

To conclude, material receives optimum grinding efficiency with %80 MFR for lower D (50) value. In addition, longer grinding duration which is 180 minutes of grinding provides the lowest D (50) values. Test results provide strong correlation between particle size and pozzolanic activity. Thus, pozzolanic reaction activity of waste material enhances by grinding process. Results show that Frattini test might be one of the effective methods to investigate the pozzolanic activity of the material. The other test methodologies which are carried out, provide supportive data results instead of indicating if material is pozzolanic. Finally, the investigation of the pozzolanic reactivity of brick wastes through the grinding process has revealed beneficial insights into the potential management of brick waste as supplementary cementitious material.

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APPENDICES

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