

**DEVELOPMENT OF STRUCTURAL SUPERCAPACITOR
USING RED MUD-BASED CEMENTITIOUS COMPOSITE
AS AN ELECTROLYTE**

BY

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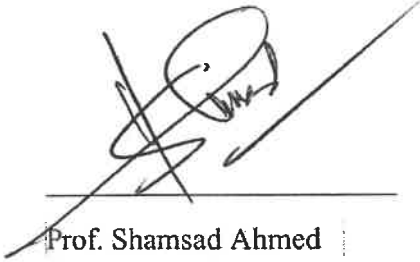
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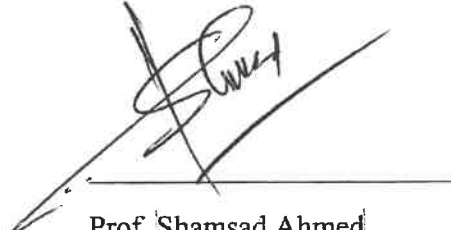


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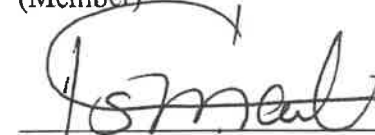
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|Dedicated to my Advisors, family and friends |

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LIST OF ABBREVIATIONS

RM	:	Red Mud
OPC	:	Ordinary Portland Cement
TEM	:	Transmission Electron Microscopy
XRD	:	X-Ray Diffraction
FTIR	:	Fourier Transform Infrared spectroscopy
EDS	:	Energy Dispersive Spectroscopy
XPS	:	X-Ray Photon Spectroscopy
XRF	:	X-Ray Fluorescence
BET	:	Brunauer–Emmett–Teller
TGA	:	Thermogravimetric analysis
DSC	:	Differential Scan-ning Calorimetry
TMA	:	Thermomechanical Analyzer
FE-SEM	:	Field Emission Scanning Electron Microscopy
ECC	:	Engineered Cementitious Composites
GGBFS	:	Ground Granulated Blast Furnace Slag
MK	:	Metakaolin

RHA : Rice Husk Ash

VOCs : Volatile Organic Compounds

MnRR1 : Mitochondrial Nuclear Retrograde Regulator 1

SEM : Scanning Electron Microscopy

ABSTRACT

Full Name : [ZAKARIA MOHAMED NOR]

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The disposal of red mud (RM), a waste material generated by the aluminum industry, remains a global environmental concern because of its high alkalinity and smaller particle size, which have the potential to pollute air, soil, and water. Recently, efforts have been made to develop a strategy for reusing industrial byproducts, such as RM, and turning waste into value-added products. The use of RM as (i) a supplementary cementitious material for construction and building materials, such as cement, concrete, bricks, ceramics, and geopolymers, and (ii) a catalyst is discussed in this review. Furthermore, the physical, chemical, mineralogical, structural, and thermal properties of RM, as well as its environmental impact, are also discussed in this review. It is possible to conclude that using RM in catalysis, cement, and construction industries is the most efficient way to recycle this byproduct on a large scale. However, the low cementitious properties of RM can be attributed to a reduction in the fresh and mechanical properties of composites incorporating RM. On the other hand, RM can be used as an efficient active catalyst to synthesize organic molecules and reduce air pollution, which not only makes use of solid waste but also lowers the price of the catalyst. The review provides basic information on the characterization of RM and its suitability in various applications, paving the way for more advanced research

on the sustainable disposal of RM waste. Future research perspectives on the utilization of RM are also addressed.

The most basic capacitor consists of two conducting plates submerged in an electrolyte and an insulating surface between them. These plates possess different charges; one is negative and the other is positive. The former acquires positive charges from the electrolyte when a voltage is applied across the capacitor, whereas the positive plate acquires negative ions. The separation and the charges in the capacitor produce an electric field, which charges the capacitor. This is because the insulating material prohibits the movement of electrons. When necessary, the two plates may instantly deliver this pair of charges after maintaining them for a lengthy period. Supercapacitors are simple capacitors with incredibly enormous charge storage capacities. The idea of supercapacitors becomes more relevant when considering the global rise in energy demand, the quick depletion of nonrenewable energy supplies, and environmental issues like climate change and global warming. For these reasons, the exploitation of unconventional renewable energy sources is of great interest on a global scale. Because of its high-power density, high specific capacitance, and improved cycle stability, supercapacitors stand out among other energy storage technologies as highly effective portable power storage units that may be used for a variety of applications, including consumer electronics and electric vehicles. One of the building materials that are thought and studied in this work to be a good candidate for supercapacitor manufacturing is bauxite residue or red mud (RM).

Structural supercapacitors (SSCs) made of building materials have been viewed as promising devices for large-scale energy storage applications because of their enormous volume and porosity. However, because it is challenging to combine both electrochemical performance with load-bearing capacity for building materials-based electrolytes, Moreover, because electrode materials still need to be explored to match the electrolytes, the shortage of desirable electrolytes and electrode materials impedes the rapid development of Structural supercapacitors. In this work 5 samples were tested and studied which are supercapacitors made of 0%, 20%, 40%, 60% and 80% replacement of sand by RM. Doing Mechanical tests such as compressive strength, split tensile strength and modulus of elasticity revealed that the optimum replacement was the 20%. So further the supercapacitor made with 20% RM replacement were investigated in order to assess the performances of the supercapacitor using Galvanostatic Charge Discharge (GCD), Cyclic voltammetry (CV), Electrochemical Impedance Spectroscopy (EIS) and Cyclic stability tests. |

ملخص الرسالة

الاسم الكامل: زكريا محمد نور

عنوان الرسالة: تطوير المكثفات الهيكلية الفائقة باستخدام مركب أسمنتي قائم على الطين الأحمر كإلكتروليت.

التخصص: الهندسة المدنية

تاريخ الدرجة العلمية: ديسمبر 2023

يظل التخلص من الطين الأحمر (RM)، وهو مادة النفايات الناتجة عن صناعة الألومنيوم، مصدر قلق بيئي عالمي بسبب قلوته العالية وحجم جزيئاته الأصغر، والتي لديها القدرة على تلويث الهواء والتربة والماء. في الآونة الأخيرة، بُذلت جهود لتطوير استراتيجية لإعادة استخدام المنتجات الثانوية الصناعية، مثل RM، وتحويل النفايات إلى منتجات ذات قيمة مضافة. تمت مناقشة استخدام RM باعتباره (1) مادة أسمنتية تكميلية للبناء ومواد البناء، مثل الأسمنت والخرسانة والطوب والسيراميك والجيوبوليمرات، و(2) كمحفز تمت مناقشته في هذه المراجعة. علاوة على ذلك، تمت مناقشة الخصائص الفيزيائية والكيميائية والمعدنية والهيكلية والحرارية لـ RM، بالإضافة إلى تأثيرها البيئي، في هذه المراجعة أيضًا. ومن الممكن أن نستنتج أن استخدام RM في صناعات الحفر والأسمنت والبناء هو الطريقة الأكثر فعالية لإعادة تدوير هذا المنتج الثانوي على نطاق واسع. ومع ذلك، يمكن أن تعزى الخصائص الأسمنتية المنخفضة لـ RM إلى انخفاض الخواص الطازجة والميكانيكية للمركبات التي تتضمن RM. من ناحية أخرى، يمكن استخدام RM كمحفز فعال لفعال لتجميع الجزيئات العضوية وتقليل تلوث الهواء، الأمر الذي لا يستفيد من النفايات الصلبة فحسب، بل يخفض أيضًا سعر المحفز. توفر المراجعة معلومات أساسية عن توصيف RM ومدى ملائمتها لمختلف التطبيقات، مما يمهّد الطريق لمزيد من الأبحاث المتقدمة حول التخلص المستدام من نفايات RM. كما يتم تناول وجهات النظر البحثية المستقبلية حول استخدام RM.

يتكون المكثف الأساسي من لوحين موصلين مغمورين في محلول كهربائي وسطح عازل بينهما. تمتلك هذه الصفائح شحنات مختلفة؛ أحدهما سلبي والآخر إيجابي. يكتسب الأول شحنات موجبة من المحلول الكهربائي عندما يتم تطبيق جهد عبر المكثف، في حين تكتسب اللوحة الموجبة أيونات سالبة. ينتج عن الانفصال والشحنات في المكثف مجالًا كهربائيًا، والذي يشحن المكثف. وذلك لأن المادة العازلة تمنع حركة الإلكترونات. عند الضرورة، قد تقوم اللوحات بتوصيل زوج الشحنات هذا على الفور بعد الحفاظ عليهما لفترة طويلة. المكثفات الفائقة هي مكثفات بسيطة ذات ساعات تخزين شحن هائلة بشكل لا يصدق. أصبحت فكرة المكثفات الفائقة أكثر أهمية عند النظر في الارتفاع العالمي في الطلب على الطاقة، والاستنزاف السريع لإمدادات الطاقة غير

المتجددة، والقضايا البيئية مثل تغير المناخ والاحتباس الحراري. ولهذه الأسباب، فإن استغلال مصادر الطاقة المتجددة غير التقليدية يحظى باهتمام كبير على نطاق عالمي. بسبب كثافة الطاقة العالية، والسعة النوعية العالية، واستقرار الدورة المحسن، تبرز المكثفات الفائقة بين تقنيات تخزين الطاقة الأخرى كوحدات تخزين طاقة محمولة فعالة للغاية يمكن استخدامها لمجموعة متنوعة من التطبيقات، بما في ذلك الإلكترونيات الاستهلاكية والمركبات الكهربائية. إحدى مواد البناء التي يُعتقد أنها تمت دراستها في هذا العمل لتكون مرشحاً جيداً لتصنيع المكثفات الفائقة هي بقايا البوكسيت أو الطين الأحمر (RM).

يُنظر إلى المكثفات الفائقة الهيكلية (SSCs) المصنوعة من مواد البناء على أنها أجهزة واحدة لتطبيقات تخزين الطاقة واسعة النطاق بسبب حجمها الهائل ومساميتها. ومع ذلك، نظرًا لأنه من الصعب الجمع بين كل من الأداء الكهروكيميائي والقدرة على التحمل للإلكترونيات القائمة على مواد البناء، علاوة على ذلك، نظرًا لأن مواد الإلكترونيات لا تزال بحاجة إلى الاستكشاف لتناسب مع الإلكترونيات، فإن النقص في الإلكترونيات ومواد الإلكترونيات المرغوبة يعيق التطور السريع للمكثفات الفائقة الهيكلية. في هذا العمل تم اختبار ودراسة 5 عينات وهي عبارة عن مكثفات فائقة مصنوعة من 0%، 20%، 40%، 60% و 80% من استبدال الرمل بواسطة RM. وأظهر إجراء الاختبارات الميكانيكية مثل مقاومة الانضغاط ومقاومة الشد المنفصل ومعامل المرونة أن الاستبدال الأمثل هو 20%. علاوة على ذلك، تم فحص المكثف الفائق المصنوع من استبدال RM بنسبة 20% من أجل تقييم أداء المكثف الفائق باستخدام تفريغ الشحنة الجلفانوستاتيكية (GCD)، قياس الجهد الدوري (CV)، التحليل الطيفي للمقاومة الكهروكيميائية (EIS) واختبارات الثبات الدوري. |

CHAPTER 1

INTRODUCTION

Red mud (RM) is a type of industrial waste produced in large quantities during the Bayer process of extracting alumina from bauxite ore. The chemical and mineral composition of the RM varies widely depending on the bauxite source and the processes involved. In general, it is composed of six major oxides: calcium oxide, silicon dioxide, iron (III) oxide, aluminum oxide, titanium dioxide, and sodium oxide, with minor amounts of other constituents. It is estimated that the global stock of RM had exceeded 5 billion tons until 2020 had.^[1] Due to the high alkalinity of RM, large-scale disposal would result in significant harmful emissions.^[2] Thus, it is necessary to investigate various alternative approaches for the efficient and sustainable use of RM. Because RM contains metal oxides in relatively higher concentrations such as aluminium oxides, iron oxides, titanium oxides, silica oxides, and hydroxides, it is a suitable industrial waste material as a low-cost adsorbent for waste gas purification and wastewater treatment applications. RM has also been widely utilized as a construction and building material, contributing towards sustainability because the process of producing ordinary Portland cement (OPC) is regarded as one of the major contributors to global carbon-dioxide (CO₂) emissions.^[3] According to various studies, the production of one ton of cement results in the emission of one ton of CO₂ into the atmosphere. Therefore, the demand for low-carbon cementitious materials is growing in the construction sector to limit the discharge of such greenhouse gases by employing low-carbon

cementations alternatives to OPC. The use of supplementary cementitious materials and alkali-activated materials, among other strategies, has been recommended in the literature as different ways to reduce CO₂ emissions during the production of cement and concrete. RM, among other waste materials, is used as a partial replacement for cement in concrete mixes, with various beneficial effects. RM serves as a strong binder material and protects the reinforcement from corrosion.^[4] As a result, it can be utilized as a partial replacement for OPC in mortar and concrete mixes. Furthermore, RM can be used as an alternative to conventional raw materials in the manufacture of bricks, which not only reduces raw material costs but is also very important in terms of sustainability. In addition, RM can be used for pigment work. The only product made from RM is pozzolanic pigment, which is utilized to color concrete through calcination at temperatures ranging from 600 to 800 °C.^[5] Another potential use for RM is in ceramics and as a corrosion-resistant material. Interestingly, the use of RM allows for the recovery of valuable elements like sodium, aluminum, and iron. Further, because RM is a mixture of oxides and hydroxides with fine particles, the idea of using it as an adsorbent was inspired. Numerous studies have documented the usage of RM for adsorbing pollutants from water or wastewater, including as phosphate,^[6] congo red,^[7] fluoride,^[8] nitrate,^[9] heavy metals,^[10] etc.

Another potential application for RM is as a catalyst, which could be a good alternative to commercially available catalysts.^[11] Noble metals and metal oxide catalysts have attracted a lot of attention for decades as an efficient catalyst for numerous industrial applications, such as pharmaceuticals, agrochemicals, paints, polymers, and others. This is because noble metals are active, selective, and easy to tune to a wide variety of catalysts. The real drawback of using noble metal-based homogeneous catalysis is its high cost, low stability,

complexities in handling, volatilization, poisoning, and sintering nature.^[1] In addition, the cumbersome preparation of the ligands, multiple steps, the use of tons of toxic organic solvents, and the chromatographic separation of the catalysts from the product are all time-consuming tasks. These catalysts have a low reusability because they are highly sensitive to air, moisture, and oxygen. In this context, immobilization of the same catalysts onto the solid supports becomes the obvious choice for enhanced stability, effortless separability, and higher cycles of reusability. Saving noble metals and reducing the cost of catalyst fabrication are both highly desirable. Transition-metal-mediated heterogeneous catalysis in this context opens up a completely new promising area for the organic functional group transformation. Transition metals, such as V, Cr, Ti, Fe, Co, Ni, Mn, Cu, Zn, and their metal oxides, such as V₂O₅, CrO₅, Fe₂O₃, Fe₃O₄, TiO₂, ZnO, NiO, CuO etc., are more stable, economical and offer ample opportunities to modify their surfaces and combine with other metals to develop bimetallic or composite systems. As a result, heat-treated RM^[4] can be a valuable asset because it contains a series of transition metals, such as Na, V, Fe, Ni, Cu, Al, Si, and their corresponding oxides,^[5] which can be used to substitute commercial transition metal or metal oxides-based catalysts for numerous organic functional group interconversions.^[6] In line with these efforts, a series of organic functional group transformations catalyzed by RM have appeared in the literature and are discussed in this review.

This thesis focuses on the characterization of RM in terms of morphological, elemental, structural, and physical properties. Furthermore, this review discusses potential applications of RM in various fields, including construction as a building material, masonry bricks, ceramics, geopolymers, and, finally, as catalysts. Its effect on the fresh and hardened characteristics of concrete has been addressed. Towards the end, future research perspectives

are proposed for expanding and improving the beneficial applications of RMs, as well as contributing towards the sustainable use of this industrial waste material. |

CHAPTER 2

LITERATURE REVIEW

2.1 Characterization of RM

Characterization is a highly useful technique to evaluate physical, mechanical, chemical, thermal, structural, and microstructural characteristics of a material. It aids in identifying advanced materials, comprehending their behavior at the micro-macro level, and understanding their impact on the environment. It is a crucial process for improving material quality and developing more advanced materials/products. With technological advancements, various cutting-edge material techniques for characterizing materials are now available. For morphological characterization, for example, the most commonly used characterization techniques are Field Emission Scanning Electron Microscopy (FE-SEM), Transmission Electron Microscopy (TEM). For chemical structure characterization, X-Ray Diffraction (XRD), Fourier Transform Infrared spectroscopy (FTIR), and Raman spectroscopy are used. For elemental composition characterization, Energy Dispersive Spectroscopy (EDS), X-Ray Photon Spectroscopy (XPS), and X-Ray Fluorescence (XRF) are used. For physical characterization, Brunauer–Emmett–Teller (BET) is widely used. For thermal characterization, Thermogravimetric analysis (TGA), Differential Scanning Calorimetry (DSC), and Thermomechanical Analyzer (TMA).

Various researchers used the aforementioned techniques to characterize the RM obtained from various sources. The sections that follow discuss the characterization studies on RM material that have been reported in the literature.

2.1.1 Morphological Characterization

Various researchers have examined the morphology, shape, and surface texture of the RM particles. For instance, Pinnock and Gordon [12] conducted SEM micrographic analysis and revealed that 70% of the RM particles are less than 1 μ m in size. Amorphous coatings of either alumina gel, amorphous iron oxide hydrate, or a combination of the two were used to adhere the RM particles to single or agglomerated particles. Deihimi et al. [13] studied the RM morphology and reported that most of the RM particles were spherical in shape, with a few flaky particles ranging in size from a few to 20 μ m. This wide range in particle size distribution was attributed to the presence of old minerals (crystalline form) as well as the formation of new minerals (amorphous form) in bauxite ore. Some of the RM mineral phases were also crystallized during the disposal process. A similar morphology of RM as spherical with a few flaky particles was reported in another study. [14] Manfroi et al. [15] used a laser grain size technique to determine the RM particle size distribution and reported that all particles were < 18 μ m in size (Fig. 1). Wu and Liu[16] reported a loose and highly porous microstructure of the RM using SEM micrographs. The particle size of grounded RM ranged between 1-80 μ m, with an average size of 26.7 μ m; however at elevated temperatures (around 1350oC), the average particle size increased to 38.2 μ m due to crystallinity enhancement. According to Yalçın and Sevinç, [17] the particle size of RM was between 5-35 μ m, with 88% of the particles being less than 10 μ m. Wang and Liu [18] tested the particle size distribution of RM and found that the RM particles ranged in diameter

from 0.8 to 50 μm , with an average size of 14.8 μm . Snars and Gilkes[19] studied the particle size distribution and surface texture of RM derived from various sources. The particle size was reported to be between 2-20 μm in most cases, with a sandy clay loam to clay texture. Fig. 1 depicts a few representative SEM micrographs of RM reported by different researchers. Meanwhile, Fig. 2 shows the particle size distribution curve of the RM reported by Manfroi et al. [15].

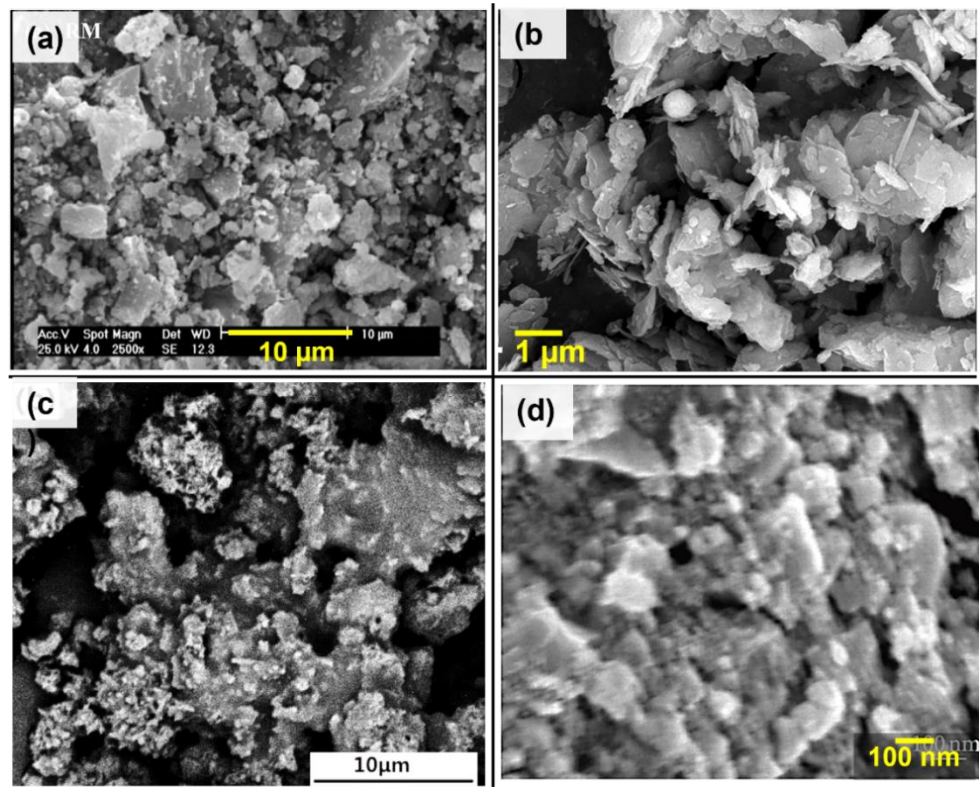


Figure 1 SEM imaging of RM (a) adapted with permission, Deihimi, et al.[13] Copyright 2018, Elsevier; (b) adapted with permission, Luo, et al.[14] Copyright 2019, Elsevier; (c) adapted with permission under the terms of the CC- BY 3.0 license, Wang and Liu[18]

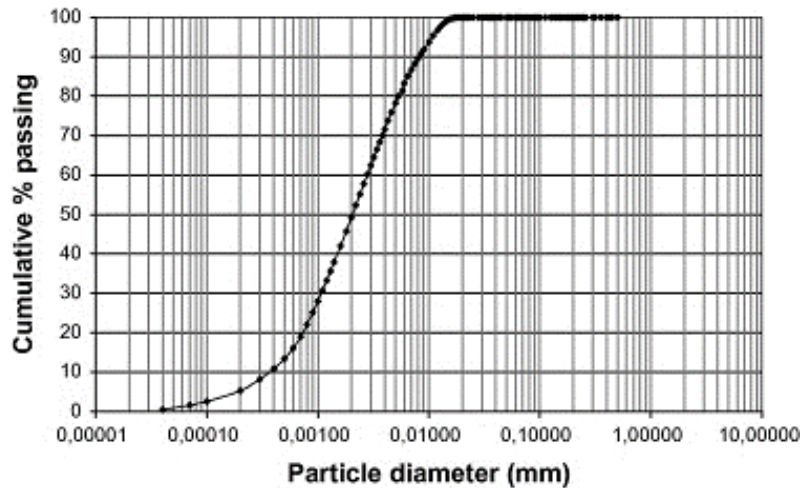


Figure 2 Particle size distribution of RM. Adapted with permission,[15]
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2.1.2 Structural Characterization

This section discusses the studies reported in the literature on structural characterizations of RM using XRD and FTIR techniques.

a. XRD analysis

XRD diffractograms of the dried RM exhibited low intensity peaks for gibbsite and boehmite, indicating that the majority of alumina was amorphous.[12] Deihimi et al. [13] used XRD to investigate the RM from the Jajarm mine (Iran) and discovered iron, titanium, and aluminum as the major components. Furthermore, it was also reported that significant amount of aluminum in RM indicates that the aluminum oxide was improperly soluble under the Bayer process operating conditions. Manfroi et al. [15] detected chantalite, calcite, quartz, cancrinite, gibbsite, and hematite phases in the XRD diffractograms of dry RM. Pera et al. [20] conducted an XRD analysis of RM and reported the existence of minerals, such as hydrogarnets, hematite, boehmite, perovskite, calcite, geothite, gibbsite, and rutile. The formation of hydrogarnets and perovskite are attributed to the occurrence of

chemical reactions between calcium carbonate and bauxite during the Bayer process. Ribeiro et al. [4] analyzed the XRD patterns of RM and reported the existence of major components, such as aluminum hydroxide ($\text{Al}(\text{OH})_3$), iron oxide (Fe_2O_3), and calcium carbonate (CaCO_3), as well as a relative amount of SiO_2 , muscovite, and $\text{FeO}(\text{OH})$. Wu and Liu[16] reported the existence of calcite, gibbsite, dicalcium silicate, hematite, perovskite, and CaO as the main mineral phases in RM using XRD diffractograms. Luo et al. [14] revealed the presence of carcanite, birnessite, calcite, hematite, perovskite, katoite, and gibbsite mineral phases using XRD analysis of RM. The crystalline phases of gismondite, goosecreekite, and epistilbite mineral compounds from the zeolite group were also discovered through XRD analysis.[21] Yalçın and Sevinç[17] conducted an XRD analysis of RM and reported the presence of phases, such as hematite ($\alpha\text{-Fe}_2\text{O}_3$), sodium aluminum oxide (NaAlO_2), sodium aluminum silicate hydrate ($1.01 \text{ Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 1.68 \text{ SiO}_2 \cdot 1.73 \text{ H}_2\text{O}$), sodium titanium oxide ($\text{Na}_2\text{Ti}_3\text{O}_7$), sodalite [$\text{Na}_4\text{Al}_3\text{Si}_3\text{O}_{12}(\text{OH})$], sodium silicate (Na_2SiO_3), iron titanium oxide (Fe_2TiO_5), and calcium silicate (CaSiO_3). Bhat et al. [22] measured the (001) basal reflections of RM through XRD analysis and reported a d-spacing of $32.76 \text{ \AA}$ at 2θ of 2.71 degrees. Using XRD analysis, Wang et al. [23] discovered the presence of four main phases in RM: calcite, geothite, hematite, and quartz. Li et al. [24] also reported the existence of major phases, such as calcite, katoite, hematite, diaspore, andradite, and muscovite. Wang and Liu[18] described the existence of perovskite, aragonite, hematite, sodium aluminate, and calcite as the main phases in the RM. Fig. 3 shows representative XRD diffractograms of RM from the literature.

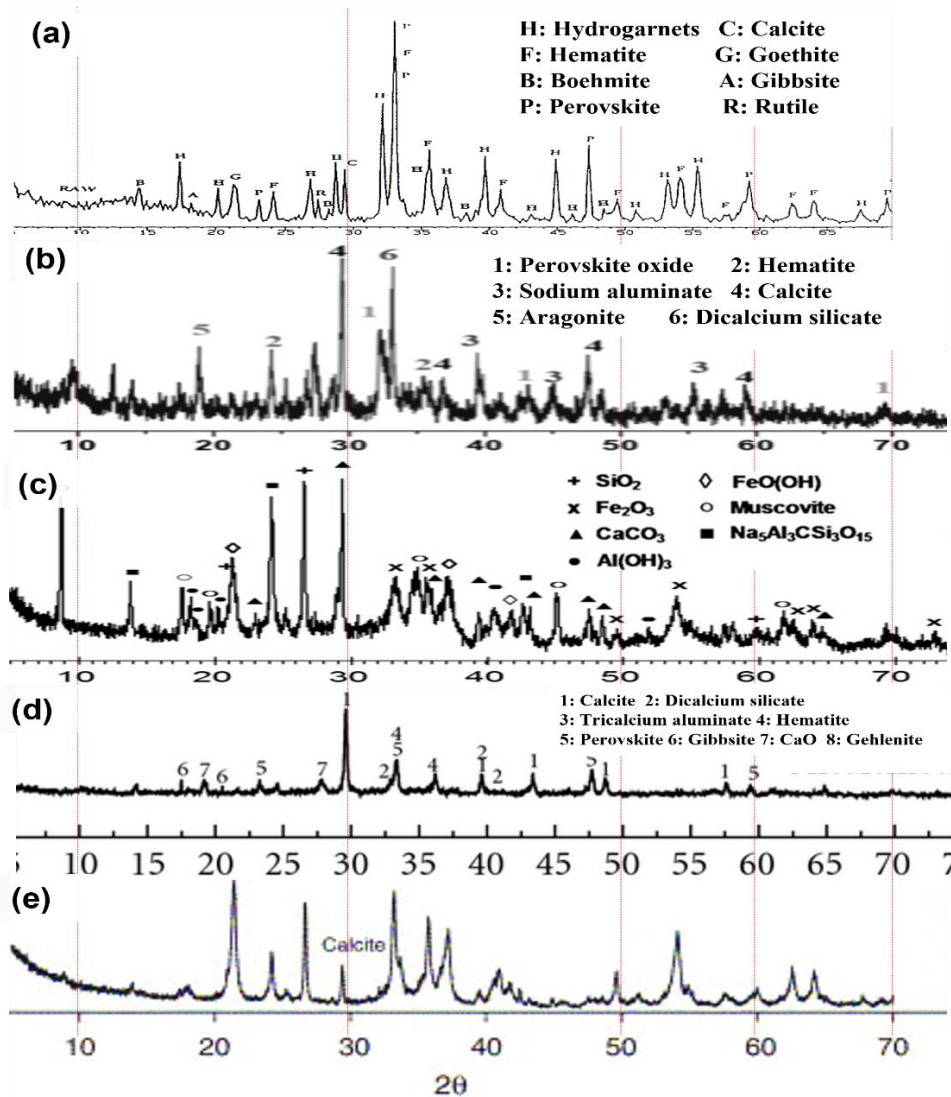


Figure 3 XRD diffractograms of RM. (a) Adapted with permission,[20] Copyright 1997, Elsevier. (b) Adapted with permission under the terms of the CC- BY 3.0 license,[18] Copyright 2012, The Authors. (c) Adapted with permission,[4] Copyright 2012, Elsevier. (d) Ad

b. FTIR analysis

Deihimi et al. [13] demonstrated the existence of physisorbed water on the RM using an FTIR spectrum with an O-H bending vibration band at 1612 cm⁻¹. Similarly, several other bands were observed indicating the presence of coordinate water molecules hydroxyl (O-H stretching vibration, 3600-3100 cm⁻¹), carbonate (1410-1470 cm⁻¹), CaCO₃ (1413 cm⁻¹), calcite (873 cm⁻¹), katoite (CaSiO₄; SiO₄ tetrahedral anti-symmetric Si-O stretching

vibrations, 919-999 cm⁻¹), aluminosilicates (Cancrinite, (Na,Ca,K)₈(Al,Si)₁₂O₂₄(SO₄CO₃)₃H₂O)) (C-O stretching vibration at 1432 cm⁻¹, Si-O-Al symmetric and asymmetric stretch at 680-600 cm⁻¹ and 1100-1120 cm⁻¹), and hematite (Fe-O stretching vibration at 460-500 cm⁻¹), etc. According to Bhat et al., [22] the raw RM exhibited the hydroxyl stretching vibration (O-H) band at 3417 cm⁻¹, Si-O stretching vibration at 993 cm⁻¹, and O-H bending at 1636 cm⁻¹ in the FTIR analysis.

c. XPS analysis

XPS is one of the potential techniques used for studying the elemental states of a material. The information on the chemical states of different elements or phases in red mud would be extremely useful in clarifying the bonding or chemical interactions or any further treatment of RM in various applications, such as cementitious matrix, geopolymer matrix, hydrothermal/acid treatment, electrochemical, catalytic applications, etc. Mudgal et al. [25] characterized the RM using XPS technique to study its interactions in the geopolymer (Fig. 4 (a-d)), and reported that the XPS characteristics peak of iron (Fe 2p_{3/2}) in RM at 711.0 eV corresponds to Fe₂O₃. Furthermore, the O1s XPS spectrum of RM revealed that 10% incorporation of RM as a replacement for fly ash in the geopolymer matrix enhanced the geopolymeric chain, i.e., the (Si-O-Na)/(Si-O-Si) bonding ratio was reduced by 1.35 times. Additionally, the spectrum revealed a higher Si-OH contribution in RM fly ash geopolymer compared to only fly ash geopolymer, resulting in enhanced geopolymer composite strength. Pande et al. [26] reported that the high convoluted O1s XPS spectrum of RM exhibited three components (Fig. 4e). The first component at ~ 530 eV represents the oxygen present in transition-metal oxides, for example iron and titanium oxides, and constitutes about 45% of the total oxygen. The second component (33% of total oxygen) at 531.6-

531.8 eV represents oxygen in alumina and metal carbonates, while the third component at 533 eV, which constitutes about 18%, corresponds to hydroxyl groups. Further, a low intensity photopeak at 536.7 eV is attributed to the Na KLL Auger peak. Zhao, et al. [27] studied the XPS survey spectrum of RM that contained major elemental peaks of O, C, Fe, Ca, and Al. Furthermore, a semi-quantitative analysis of the Fe deconvoluted XPS spectrum indicated that the Fe_2O_3 at 810.7eV: Fe_2O_4 at 709.2 eV ratio was around 2.6. This information was further utilized in an absorption study with RM. Similarly, other studies have used the XPS technique to characterize the RM to obtain valuable chemical information in order to optimize and improve the performance of RM applicability.^[28] Fig. 4 depicts representative RM XPS patterns.

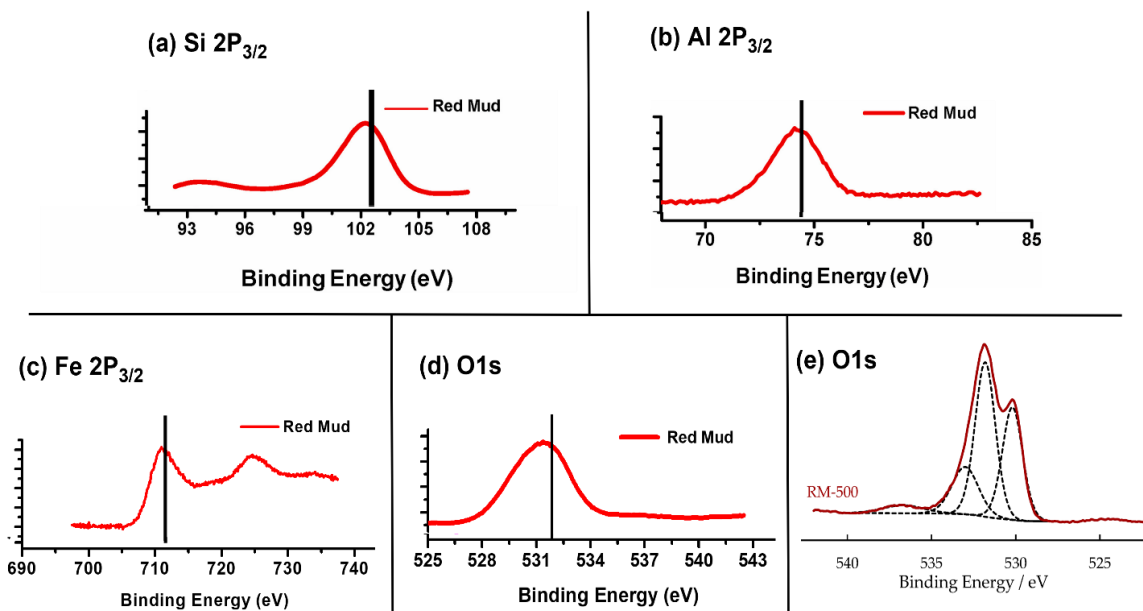


Figure 4 XPS pattern of RM. (a-d) Adapted with permission,[25] Copyright 2021, Elsevier. (e) Adapted with permission under the terms of the CC- BY 4.0 license,[26] Copyright 2012, The Authors.

2.1.3 Physical Characterization

a. BET analysis

Deihimi et al. [13] performed a BET analysis of RM and found that it has a specific surface area of 26.75 m²/g. Pera et al. [20] reported that the BET surface area of the RM was approximately 10.7 m²/g with a specific gravity of 3.05. Park and Jun[29] conducted N₂/77 adsorption, surface area, pore volume, and surface free energy studies of RM. The RM exhibited a Type-II, N₂ gas adsorption isotherm with a specific surface area of 30 m²/g and a total pore volume of 0.1047 cm³/g, while the surface free energy parameters were γ_s (55.4), γ_s^L (39.1), γ_s^+ (2.27) and γ_s^- (28.98).

2.1.4 Thermal Characterization

a. Thermogravimetric and differential thermal (TG-DT) analysis

Wu and Liu[16] used the TG-DT analysis to demonstrate a continuous weight loss distribution in the RM between 50 and 1450°C, as shown in Fig. 5. The TGA curves indicated a mass loss of 8.26% due to chemically bound and physically adsorbed water at temperatures ranging from 50 to 550°C, and a mass loss of 21.81% due to calcite decomposition at temperatures ranging 550 to 900°C. The DTA endothermic peaks of RM revealed the decomposition of gibbsite (300-550°C), calcite (600-800°C), and the formation of tricalcium aluminate and gehlenite (800-900°C). Luo et al. [14] investigated the thermal performance of RM and reported that the physically adsorbed and chemically bounded water evaporated at temperatures ranging from 25 to 100°C. Gibbsite, birnessite, and calcite decomposed at temperatures ranging from 120 to 310°C, 400 to 550°C, and 550 to 700°C, respectively, while cancrinite and katiote decomposed at temperatures between 780-950°C. Yalçın and

Sevinç^[17] reported that the surface adsorbed water on the RM dehydrated at temperatures between 240-350°C, while chemically bounded water evaporated at temperatures ranging from 460 to 590°C. Furthermore, the SO₃ decomposition occurred at temperatures around 700-780°C, while the burning of organic matter and release of CO₂ occurred at temperatures around 370°C and 850-940°C, respectively. Bhat et al. ^[22] reported that the RM is thermally stable up to 267°C, with only 11% weight loss observed at 600°C with 88% residue, indicating its good thermal stability. Wang and Liu^[18] evaluated the thermal performance of RM and discovered a continuous weight loss at temperatures between 25-900°C. The free water evaporated at temperatures ranging from 25 to 150°C, while the CaCO₃ decomposed at 625°C. Fig. 5 depicts a set of representative TG-DT analysis curves of RM from the literature. Table 1 summarizes the details of RM characterization reported in the literature.

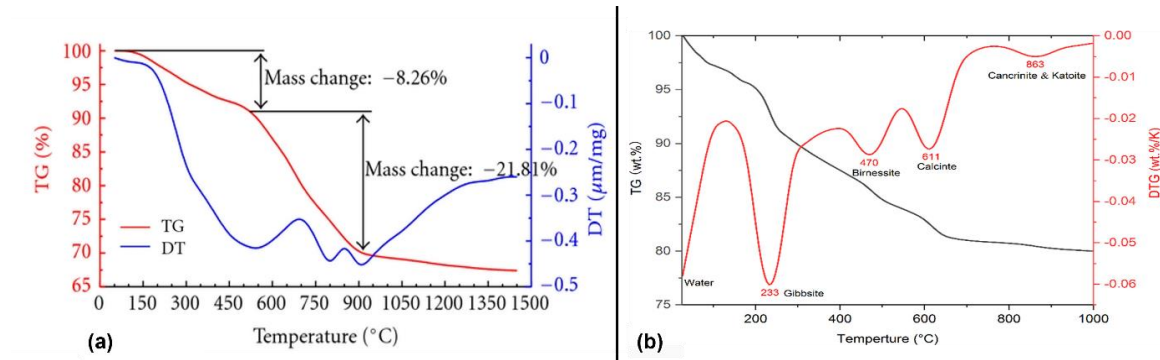


Figure 5 TG-DT analysis of RM (a) Adapted with permission under the terms of the CC license,[16] Copyright 2012, The Authors. (b) Adapted with permission,[14] Copyright 2019, Elsevier.

Table 1 Characteristic details of RM obtained from different sources

Source of RM	Particle size and shape	BET surface area (m ² /g)	Pore volume (cm ³ /g)	Specific gravity	pH	Year	Ref.
Surface of a pond (Operation B)	70% < 1µm	-	-	-	-	1992	[12]
Jajarm mine, Iran	Few to 20 µm, spherical and few flaky type	26.75	-	-	-	2018	[13]
Henan, China	0.1-1000 µm	-	-	-	-	2019	[14]
Minas Gerais	< 18 µm	-	-	-	-	2014	[15]
Guizhou, China	26.7 µm	-	-	-	-	2012	[16]
Konya, Turkey	5-35 µm	-	-	-	-	2000	[17]
Guizhou, China	0.8-50 µm diameter	-	-	2.64	-	2012	[18]
Australian and oversea	-	15-30	-	-	8.4-12.6	2009	[19]
Gardened, France.	Finer than 100 µm	10.7	-	3.05	-	1997	[20]
ALCOA Brazil	Average 8 µm, max. 40 µm	20.27	-	2.90 kg/dm ³	12.95	2012	[4]
Shanxi Province, China	76% passing through 45 µm sieve	-	-	-	-	2016	[21]

Western Power, Australia	-	21.0	0.058 (mL/g)	-	12.0	2005	[23]
Korea Chemical Co	80 nm	30.0	0.1047	-	11.20	2005	[29]

According to the studies presented in this review article on RM characterization, the RM particle size ranges from 80 nm to 1 mm, the BET surface area ranges from 10.7 to 30.0 m²/g, and the total pore volume ranges from 0.058 to 0.1047 cm³/g. The specific gravity of the RM ranges from 2.64 to 3.05, and the pH ranges from 8.4 to 12.95.

2.2 Impacts of RM on the Environment

RM is one of the toxic solid wastes produced in alumina plants during the bauxite alumina extraction process.[30] In general, every ton of alumina produced yields 1 to 1.5 tons of RM.[30b] For instance, in Saudi Arabia, approximately 230 million tons of bauxite ore were processed in 2016. RM contribute significantly to environmental contamination due to its high alkalinity (pH>12) and heavy metal content.

Alkaline solution and RM slurry are frequently leaked into the soil or underground water from RM landfills and pipelines.[31] Additionally, dams used for RM storage are large and extremely expensive to build and maintain.[30b] An alumina plant with a capacity of 1 million ton/year needs about 1 km² of land every five years to effectively dump RM, which would be logistically challenging and expensive for the alumina manufacturing industry.[32] Over 3.0 billion tons of RM are currently estimated to be stockpiled on land, with a worldwide yearly growth rate of about 120 million tons. After drying, the RM-covered

soil can no longer be used for construction or cultivation because the toxic compounds in it leach into the soil, pollute it, and contaminate groundwater.[33]

Because RM is a very fine powder, its improper disposal would have a negative impact on the surrounding air, land, and water environments.[34] In both drier and warmer climates, inhaling caustic chemicals from emission sources poses a serious threat to human health. The most severe effects of RM waste exposure include land consumption, air pollution, corrosion of building exteriors, water and soil contamination, radioactive hazards, and ecosystem destruction (Figure 6). The construction industry is one of the fastest growing industries, and RM can be utilized as an alternative construction and building material, mitigating the hazard posed by RM waste.

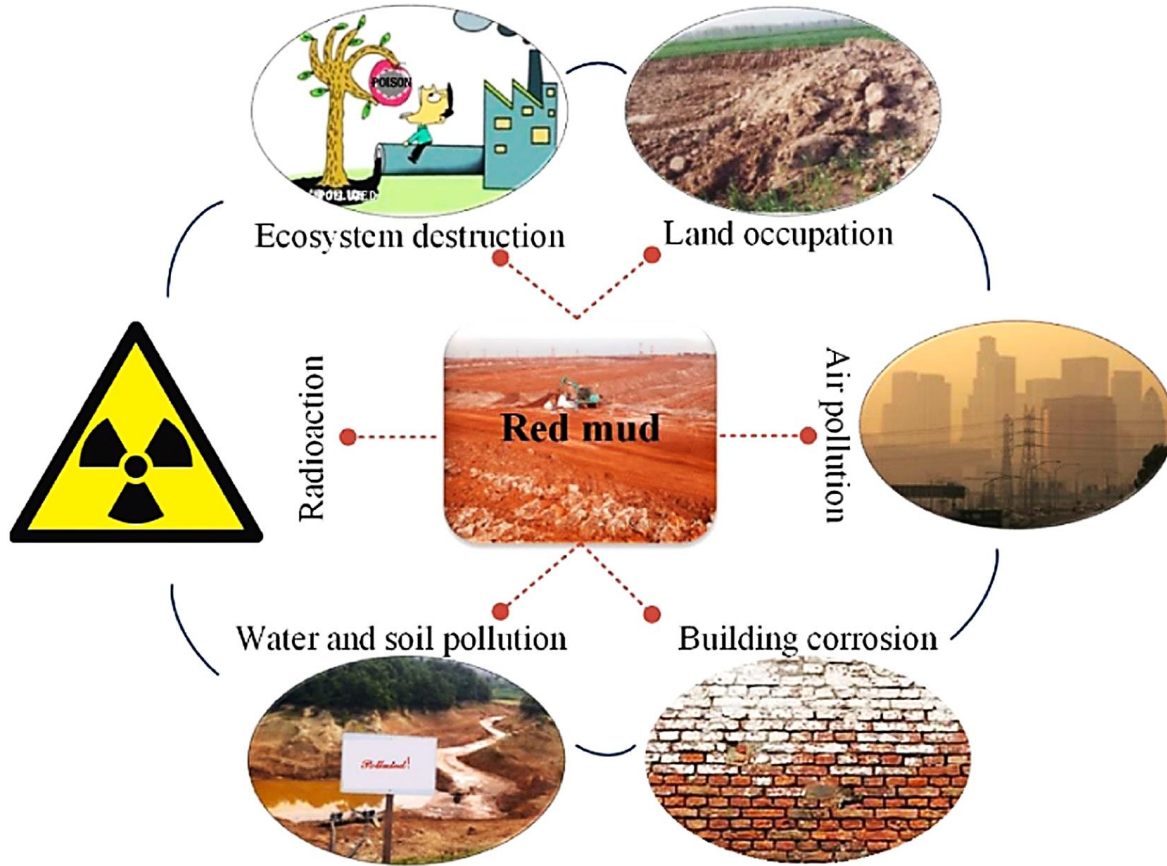


Figure 6 RM impacts on the environment

2.3 Impacts of RM on the Environment

Over the years, RM has been widely utilized in different applications, such as composting,[36] wastewater treatment,[37] stabilization material,[38] metal recovery,[30b] phosphate removal,[39] and extraction of rare earth metal.[40] Moreover, 55% of RM applications are directed to the construction and building materials sector (Fig. 7),[30a, 41] with civil and construction accounting for 34%, ceramic materials accounting for 13%, and steel making and slag additives accounting for 8%. Many proposals have been advanced for the utilization of RM in the manufacturing of masonry bricks,[42] ceramics,[43] cement and concrete,[3-4,21,32,44] composite materials,[44q,45] aggregate,[46] pigments,[20,47] geopolymers,[25,30a,41a,42c,48] painting,[49] and road construction and pavement.[50]

Therefore, the best RM application perspective is in the building and construction industry (see Figure 6), because it allows for greater RM consumption in the most diverse applications while remaining a low-cost alternative material. This section focuses on the application of RM in the development of cement and concrete, masonry bricks, ceramics, and geopolymer.

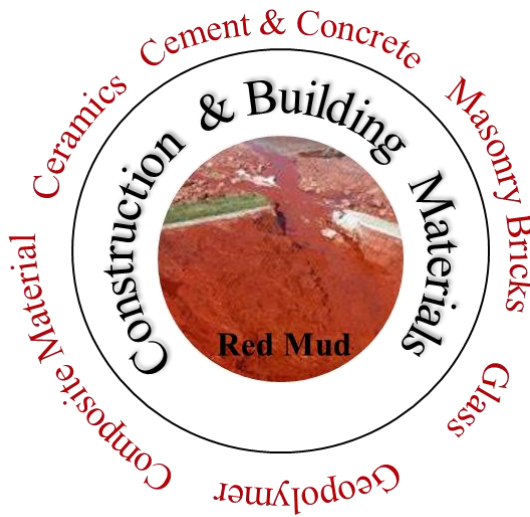


Figure 7 Applications of RM in construction and building materials sector

2.4 Applications of RM in cement and concrete

RM is a waste material with a density of 2187 kg/m^3 and a particle size distribution resembling that of fly ash. It has frequently been used in the production of concrete by replacing a portion of the OPC. This section summarizes and discusses the fresh and hardened properties of RM-based concrete, as well as the durability aspects.

Anirudh et al. ^[44 a] examined the mechanical properties and microstructure characteristics of RM-based cement mortars in which RM replaced OPC by weight percentages of 5, 10,

15, and 20%. The test results showed that the RM-based cement mortar performed better at 10% replacement. The addition of RM improved durability, and microstructure analysis revealed that RM-based cement mortar was denser than OPC-based cement mortar. This is attributed to the alkalinity of RM that aids in the development of good pozzolanic reactivity, as well as to the high specific surface area of the RM particles, which significantly contributes to the cement hydration process. When OPC is replaced with RM, water absorption is reduced by up to 20%. After 28 days of curing, RM-based cement mortar showed a reduction in water absorption by 18% (RM0) to 9% (RM20) when compared to OPC-based cement mortar. Senff et al. [44] studied the impacts of RM on the hardened properties of cement mortar at two different water to binder (w/b) ratios. As shown in Figs. 8-10, increasing the w/b ratio from 0.47 to 0.58 had a negative impact on apparent porosity, water absorption, and compressive strength. Figure 11 depicts the compressive strength results of RM-based mortar versus cement-based mortar. It can be concluded that the inclusion of RM by more than 10 wt.% caused a greater reduction in strength compared to the mortar without RM, and that this reduction can be mitigated by lowering the w/b ratio. After 360 days of curing, for example, the compressive strength of RM-based mortar (20% RM) was 13-46% lower than that of cement-based mortar. It is also worth noting that at a higher w/b ratio, the strength reduction was greater at the same RM content (see Figure 11).

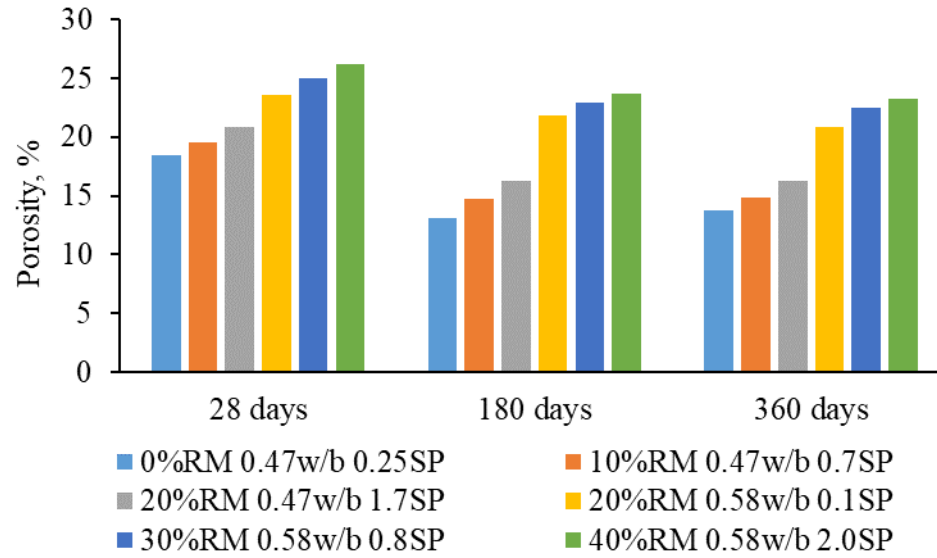


Figure 8 Apparent porosity of RM-modified cement mortar (adopted from [44r]).

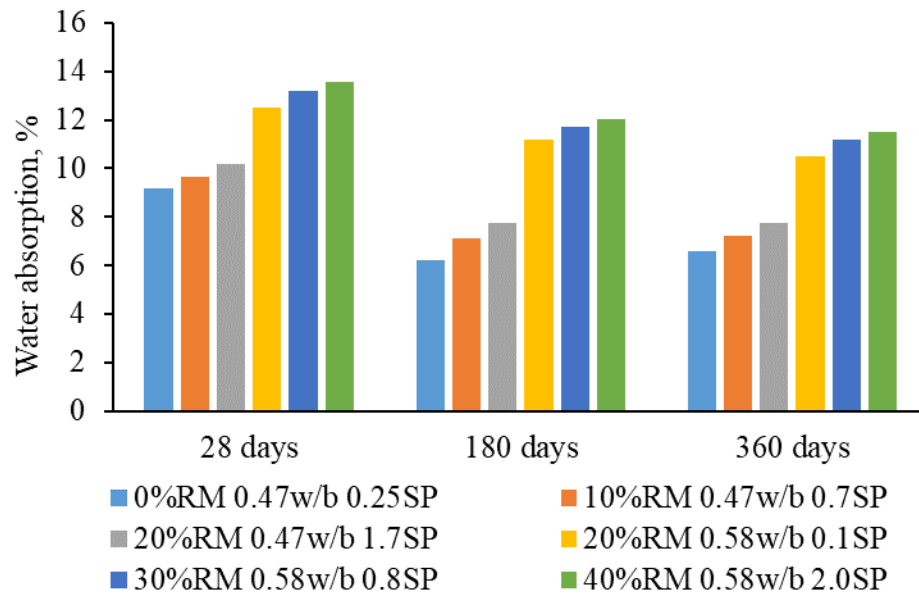


Figure 9 Water absorption of RM-modified cement mortar (adopted from [44r]).

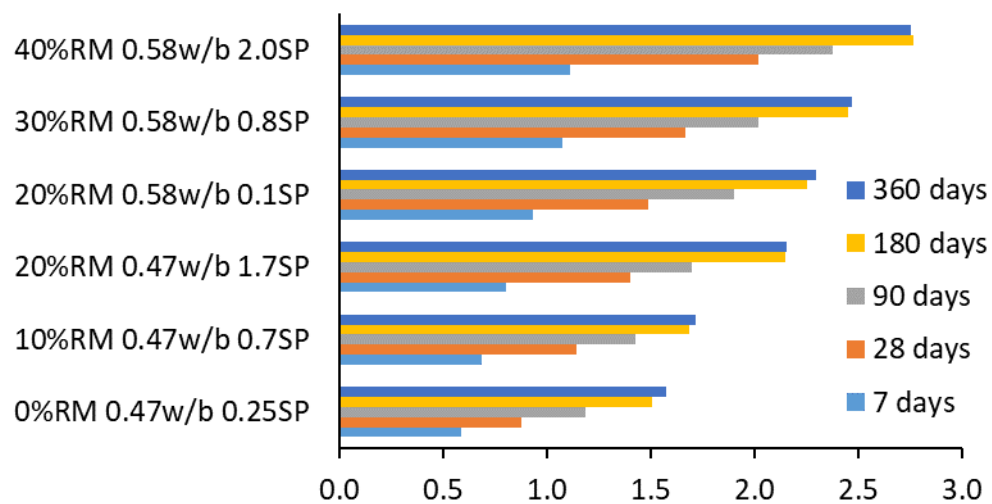


Figure 10 Shrinkage of RM-modified cement mortar (adopted from [44r]).

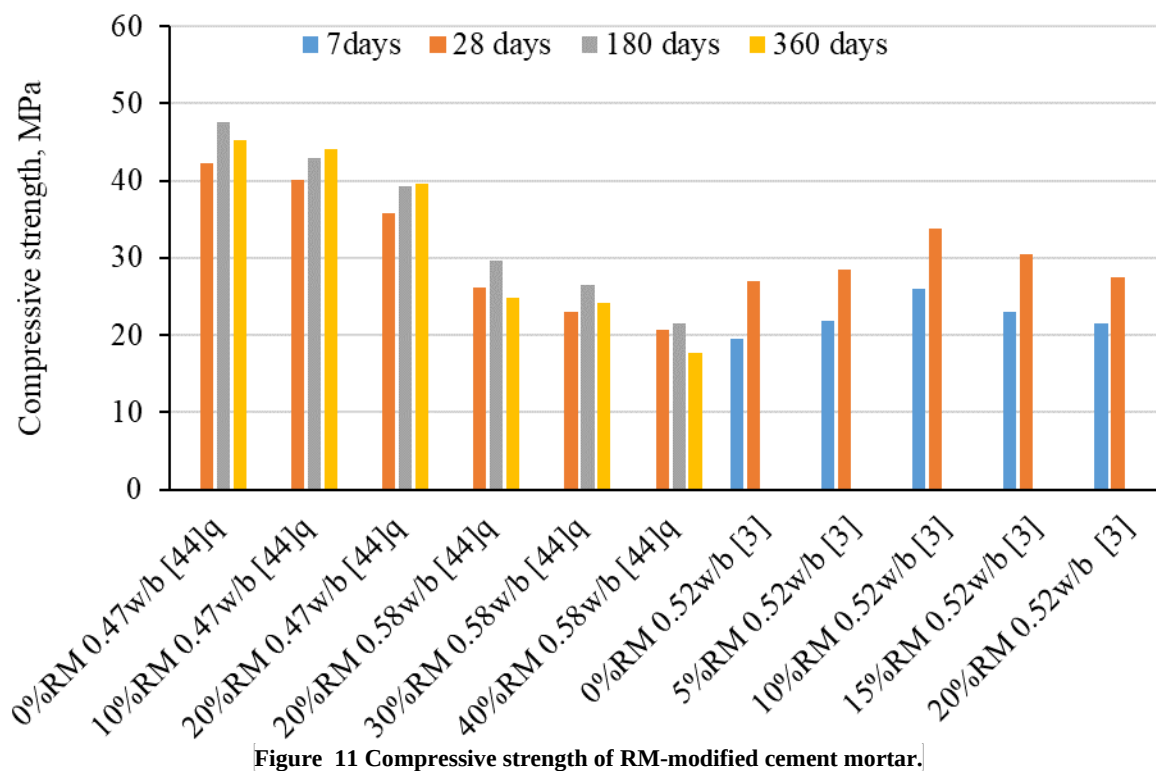


Figure 11 Compressive strength of RM-modified cement mortar.

In addition, the RM could be used as a cementitious material in self-compacting concrete (SCC) to substitute cement and fly ash. Karimipour et al. [44d] examined the mechanical strength of SCC with 0.2% steel fiber using RM as a partial substitute for OPC. In addition to the filler effect of marble slurry powder (MSP), test results indicated that 2.5% RM was the optimal substitute for attaining the highest compressive and splitting tensile strengths of SCC. Similar findings were obtained for workability compared to other research in which workability was reduced with increasing RM dosage owing to its high surface area and porosity compared to OPC. However, the reduction in SCC flowability was addressed by the addition of marble slurry powder. Moreover, combining 2.5% MSP and 5.0% RM increased the slump by 2.0 and 1.5%, respectively.

Liu and Poon^[21] evaluated the practicability of using RM as a pozzolanic material to replace 10, 20, 30, and 40 wt.% of the fly ash in SCC by studying its fresh and hardened properties. According to the study, replacing 20 wt.% of OPC in SCC with RM improves compressive and splitting tensile strengths as well as modulus of elasticity. Additionally, internal curing of RM admixed in SCC reduced drying shrinkage. This demonstrates the feasibility of using RM in SCC. More super plasticizer was required to achieve the target flow value at higher RM dosages. This could be because the porous RM absorbs more water, leaving less water available for the slump flow. Overall, the slump flow and hardened density decreased slightly while considerable reduction in segregation was noticed with increase in RM content. The fact that the RM had a lower specific gravity than fly ash was probably what caused the density reduction. However, the porosity of the mixture increased significantly at 40% RM replacement. Adding 30% and 40% RM to the SCC mix-

ture increased the compressive strength by 8% and 9%, respectively, over the control sample at 56 days due to the internal curing provided by the RM. Furthermore, the elastic modulus gradually increases to 43 GPa as the RM content increases from 0% to 30%.

Tang, et al. [44j] examined the effect of RM as a replacement for fly ash on the fresh and hardened characteristics of SCC. According to the study, RM-based concrete requires a high dosage of superplasticizer to meet the SCC requirements. The findings demonstrate that the mechanical strength of SCC improved marginally as the RM dosage increased. As shown in Fig. 12, the SCC with 50% RM had the highest compressive and tensile strengths, as well as the highest elastic modulus (34.34 GPa). Furthermore, the interfacial transition zone between the aggregate and cement paste was reportedly improved, which accounts for the slight improvement of RM-based SCC. Ghalehnovi et al. [44k] studied the effectiveness of RM addition into white cement-based SCC for architectural applications. The findings confirmed that RM has no significant effect on concrete performance, with most differences in results being less than 5% when compared to the control SCC. On the other hand, the elastic modulus of the mix with 7.5 wt. % RM decreased dramatically. Furthermore, incorporating RM into SCC can improve resistance against sulphate attack by about 27% in terms of weight loss. This is due to the pozzolanic reaction caused by RM, which altered the physical and chemical microstructure of the concrete, resulting in improved sulphate attack resistance. Additionally, using RM can assist in producing green-colored concrete with qualities comparable to those of current white cement-based concrete. Based on earlier research, the findings indicate that the RM has pozzolanic activity comparable to that of fly ash, improving the strength of SCC after 56 days of curing, especially at the

content of 20 to 50 wt.% (Figs. 12 and 13). Overall, it can be concluded that substituting fly ash by 10–40% of RM in the production of SCC is technically and practically possible.

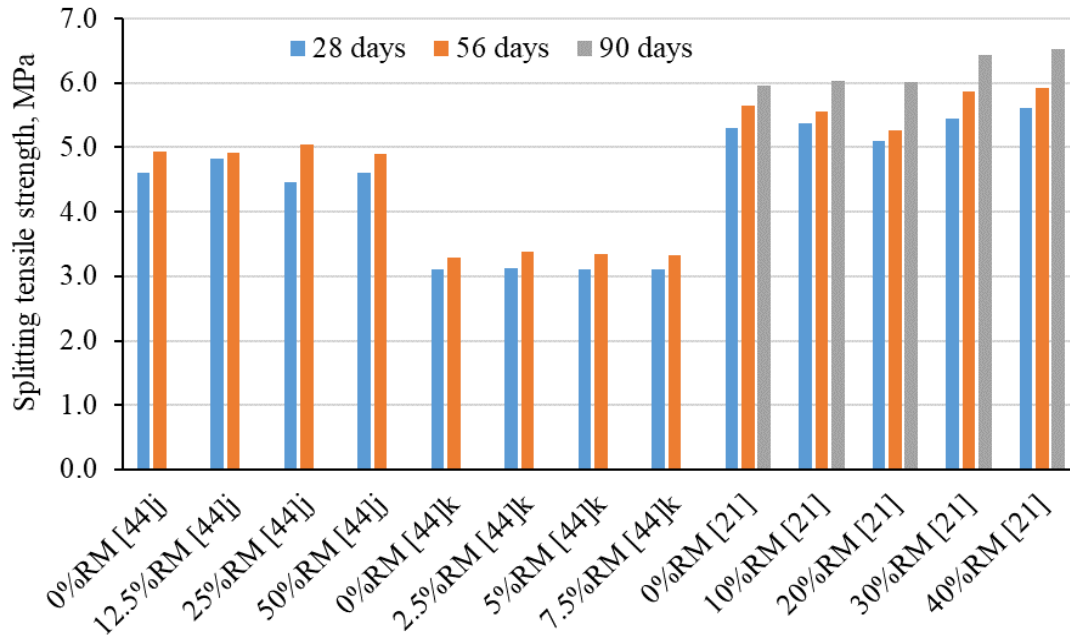


Figure 13 Splitting tensile strength of RM-based SCC.

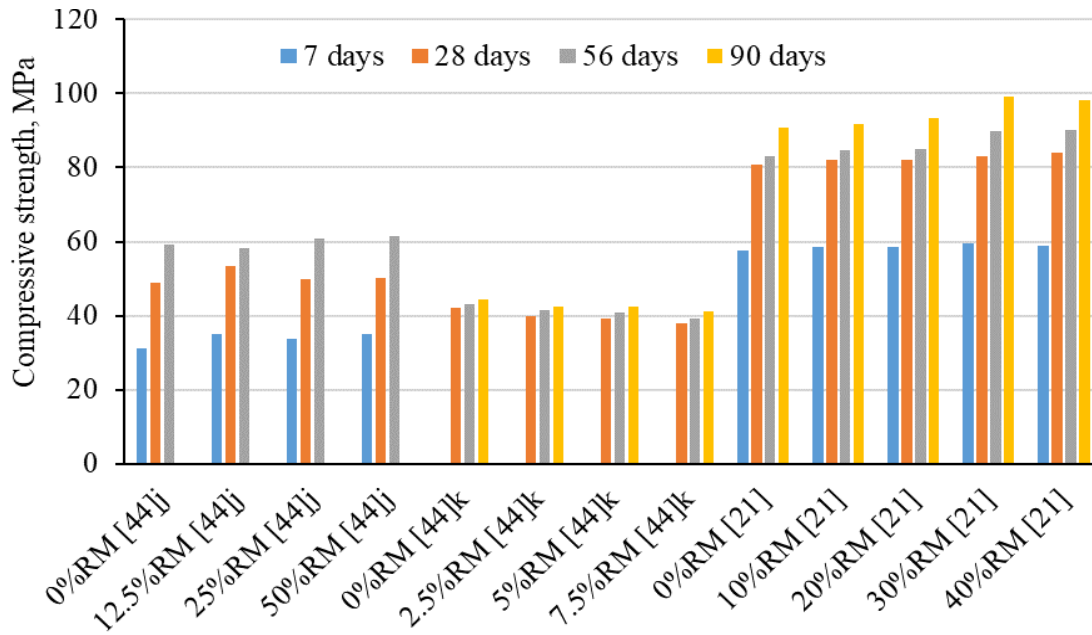


Figure 12 Compressive strength of RM-based SCC.

RM has also been added to the production of normal concrete as a partial substitute of OPC. Chavan et al. [44b] studied the characteristics of concrete containing 0 to 20 wt.% RM as a replacement for OPC. The study considered a concrete mixture of M30 grade and measured the flow, mechanical properties, as well as the chloride permeability at 28 days. The results showed that maximum mechanical strengths (see Fig. 14), superior chloride permeability, and a better microstructure than conventional concrete when OPC was partially replaced with 12 wt.% RM. The addition of RM diminished the workability of concrete because RM increases water absorption due to its higher porosity, fineness, and volume.^[51] Viyasun et al. [44e] incorporated 10, 20, and 30 wt.% of RM as a replacement of OPC in the normal concrete and examined strengths and durability. According to the findings, 20% of the weight of OPC can be replaced with RM to increase the mechanical properties of concrete (Figure 14). In terms of its durability, 30 wt.% RM replacement in OPC concrete results in a lower water absorption than conventional OPC concrete. It was also discovered that when conventional concrete was compared to RM-based concrete, the surface resistivity value was a little higher. Kumar et al. [44f] replaced the cement content in fly-ash concrete with RM ranging from 0% to 24 wt.%, as well as 8 wt.% fly ash. The study concluded that mechanical strengths increased with increasing RM content when compared to OPC-based concrete, with 20 wt.% being the optimal value of RM to replace OPC.

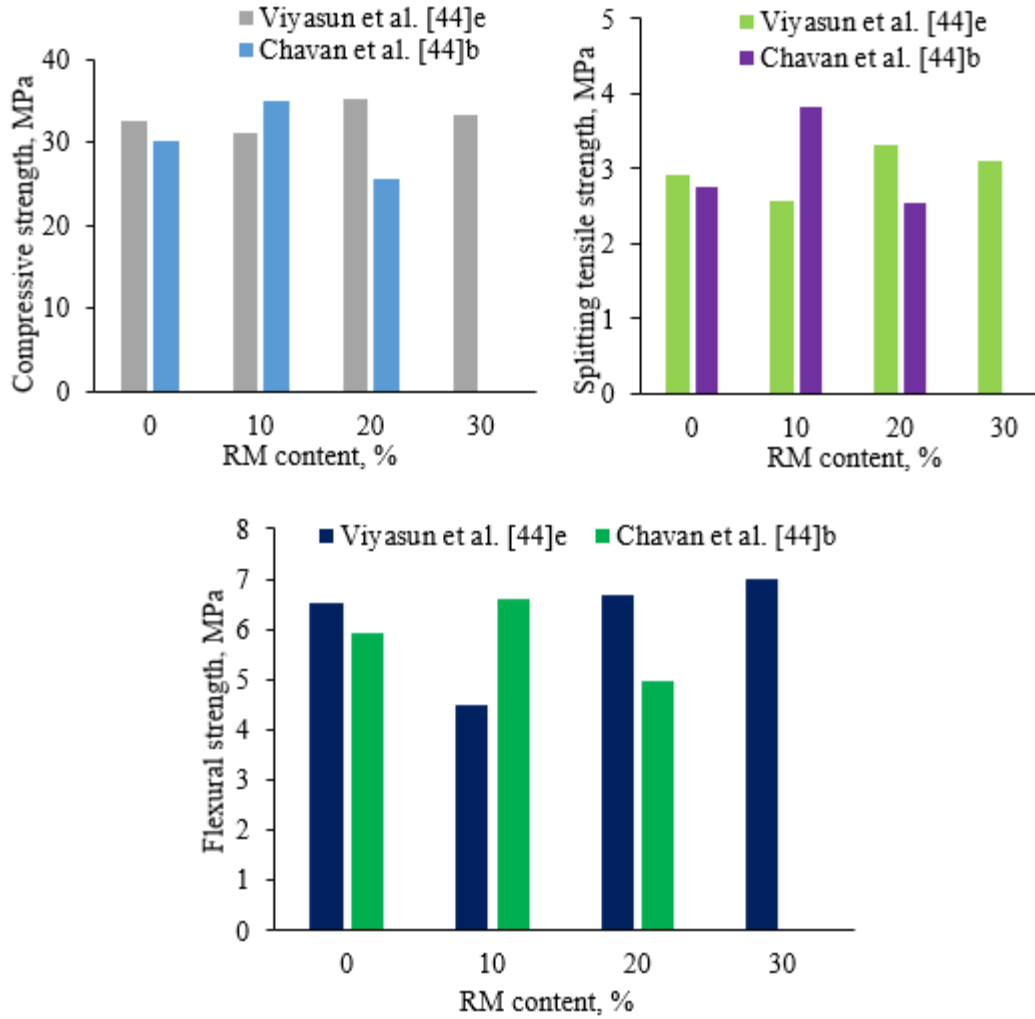


Figure 14 Mechanical strengths of RM-based normal concrete at 28 days.

The inclusion of RM significantly enhanced the water resistance of magnesium oxysulfate (MOS) cement from 0.48 to 0.68 based on the softening coefficient value, and the compressive strength increased from 24.1 MPa to 33.3 MPa after 28 days of curing (Fig. 15).[44g] This significant increase can be attributed to the amorphous gel produced by the RM and MOS cement, which compacted the cement paste matrix. It is concluded that the RM has a promising potential for use as a magnesium cement modifier to improve the

properties of building materials. Chen et al. [44m] also explored the effects of RM inclusion on the properties of gangue-cemented paste backfill (GCPB) and found that substituting 25 wt.% OPC by RM would yield the same strength and a 23% cost savings for the GCPB. RM has the ability to create new hydrates with cementitious properties as well as fill in micropores to create a denser microstructure and thus improve the mechanical properties and water resistance of the mortar at all curing ages.

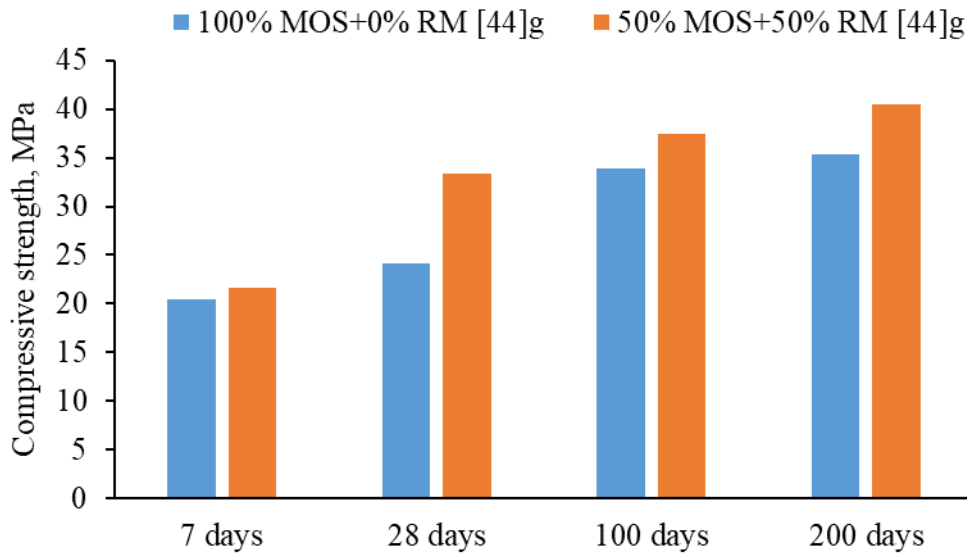


Figure 15 Compressive strength of pure MOS cement and with 50 wt.% RM.

Studies were also carried out on the use of RM as a substitute of OPC for producing lightweight concrete and high-performance concrete (HPC). Song et al. [44h] concluded that the utilization of RM as a replacement of OPC in autoclave aerated concrete (AAC) may adversely affect its workability and compressive strength. This negative effect was attributed to low activity and small particle size of the RM, which slowed the gas production process within AAC, resulting in higher thermal conductivity. Furthermore, AAC with a higher percentage of RM have a poor pore structure and high tobermorite content, which

may contribute for its lower strength. The only option to compensate for this adverse effect is to limit the RM content in ACC to 22.5 wt.% of the fly ash content and modify the water to solid ratio. Nikbin et al. [44i] investigated the compressive, splitting, and flexural strengths, as well as the elastic modulus, of lightweight concrete (LWC) incorporating RM as a partial replacement for OPC and discovered that increasing the RM dosage reduced the above-mentioned properties by up to 29.5%, 35%, 28.4%, and 26%, respectively, when compared to the LWC without RM. However, the cumulative energy demand, potential for global warming, and major air pollutants associated with LWC containing 25 wt.% of RM were reduced. Hou et al. [44c] evaluated the performance of ultra-high-performance concrete (UHPC) that incorporated 40 wt.% RM as a replacement for OPC. RM was found to reduce the workability and mechanical properties of UHPC. The increase in adsorbed water caused by the increased specific surface area of RM decreased workability. In contrast, the addition of RM significantly enhanced the early age durability of UHPC because RM possess the ability to accelerate the early hydration process and has a filling effect.^[52] Due to high alkalinity of the RM (pH=11), the initial and final setting times are shortened by 94% and 93%, respectively. Additionally, as the amount of RM increases, the micro-pore structure becomes coarser. However, the study concluded that the low energy consumption, low radiation risk, and low cost of RM-based UHPC provide a concept for the development of sustainable building materials, which encourages its use for environmental protection. Additionally, RM has been demonstrated as a potential concrete additive for corrosion prevention by reducing the corrosion potential and rate of chloride migration while raising its electrical resistivity.[4] Kan et al. [44q] incorporated RM as a total replacement of fly ash to produce engineered cementitious composites (ECC). The findings showed that all the

investigated mixes with various RM ratios can achieve excellent tensile strain capacities, with a maximum strain up to 7.43% at 40% RM replacement. RM can be used as a free raw material in the manufacture of cement without affecting the quality of the produced cement.[32] A summary of the fresh and mechanical properties of cementitious composites containing RM is listed in Figure 2. It can be concluded that the use of RM up to 20 wt.% as a replacement for OPC in the manufacture of cementitious materials was the most suitable addition

Table 2 Properties of cement-based materials containing RM (the results shown are at 28 days curing age unless otherwise stated).

No.	Type of cementitious system	w/c ratio	RM replacement of OPC (wt. %)	Physical/fresh properties				Mechanical properties			Ref.
				Slump flow dia. (mm)	Density (kg/m ³)	Porosity (%)	WA (%)	CS (MPa)	STS (MPa)	FS (MPa)	
1	Magnesium oxysulfate cement (MOS)	0.23	Mass ratio of RM to Cement is 1:1	-	↑ from 1984 to 1904	↓ from 13.2 to 9.8	↓ from 5.9 to 3.7	↑ from 24.1 up to 33.3	-	-	[44g]
2	Gangue-cemented paste backfill (GCPB)	-	0, 0.2, 0.25, 0.33 and 0.5%	-	-	-	-	↑ from 0.84 to 0.95 @0.25%	-	-	[44m]
3	Magnesium phosphate cement mortar	0.2 (w/b)	0, 10, 20, 30, and 40%	250 – 272.5 @ 20% and 250 @ 40%	-	5.46 @ 0% 2.29 @ 20% 3.2 @ 40%	-	↑ from 84 to 90.2 @20% and ↓ to 75.6 @ 40%	-	↑ from 11.9 to 12.5 @20% and ↓ to 10.5 @ 40%	[44n]
4	Cement mortar	0.52	5, 10,15, and 20%	-	-	-	↓ up to 9% at	↑ by 26.44% and 23.68% of strength at 7 & 28 days of curing at 10% replacement	-	-	[44a]
5	Cement mortar	0.47 and 0.58	0, 10, 20, 30, and 40%	↓	-	↑ from 18.5 to 26.2	↑ from 9.2 to 13.6	↓ from 42.2 to 40.1 @10% and ↓ to 20.7 @ 40%	-	-	[44r]
6	Self-compacting concrete (SCC)	0.5	10, 20, 30, and 40% replacement of fly ash.	↓ 750 - 705	↓ 2393 - 2361	↑ 7.8 - 12.2	-	↑ at longer curing age up to 90.1 (9% higher than the control at 56 days)	↑ 5.3- 6.5	-	[21]

7	Self-consolidation concrete (SCC)	0.45	0, 2.5, 5, and 10%	Ranged between 404 to 685	-	-	-	Ranged between 37 to 48	Ranged between 6.1 to 7	-	[44d]
8	Self-compacting concrete (SCC)	0.36 (w/b)	12.5, 25 and 50%	↓ 540 (0%) 502 (12.5%) 467 (25%) 490 (50%)	-	-	-	↑ 59.2 to 61.4 @ 50 wt.%	↑ 4.6 to 4.8 @ 12.5%	-	[44j]
9	Self-compacting concrete (SCC)	0.475	2.5, 5 and 7.5% as replacement of cement and 25, 50, and 75% as filler	↓ from 800 to 770	↓ from 2289 to 2279	-	↑ from 2.1 to 2.45	↓ from 42.2 to 38.0	= from 3.1 to 3.11	-	[44k]
10	M30 concrete	0.44	0, 2, 4, 6, 8, 10, 12, 14, 16, 18, and 20%	↓ 110 - 60	-	-	-	↑ 30.1 to 35 @ 12 wt.%	↑ 2.75 to 3.89 @ 12%	↑ 5.9 to 7 @ 12 wt.%	[44b]
11	M30 concrete	0.45	10, 20 and 30%	-	-	-	↓ 1.6% to 0.9%	↑ 32.6 to 35.3 @ 20 wt.%	↑ 2.8 to 3.75 @ 20 wt.%	↑ 6.5 to 7 @ 20 wt.%	[44e]
12	Fly ash-based concrete	0.57	8, 16, and 24%	-	-	-	-	↑ 34 to 39 @ 8 wt.% and ↓ to 35 @ 24 wt.%	↑ 2.75 to 3.8 @ 8 wt.% and ↓ to 2.9 @ 24 wt.%	↑ 3.45 to 3.9 @ 8 wt.% and ↓ to 3.6 @ 24 wt.%	[44f]
13	Autoclaved aerated concrete (AAC)	0.6 (w/s)	7.5, 15, 22.5, 30, 37.5, and 45% replacement of fly ash.	↓	↑ from 630 to 700 @ 158 °C	↓	↓	↓ 4.4 (control), 2.11 (22.5%), and 0.75 (45%) @ 158 °C	-	-	[44h]

14	Lightweight concrete	0.5	5, 10, 15, 20 and 25%	-	↓ from 1789 to 1685	-	↑ from 3.1 to 4.0	↓ from 29 to 20.5	↓ from 2.9 to 1.9	↓ from 8.1 to 5.9	[44i]
15	Ultra-high-performance concrete (UHPC)	0.21 (w/b)	20, 40 and 60%	↓ 260 - 100	-	↑ 11.9 – 24.9	-	↓ 159.7 (control), 135.9 (20%), 132.2 (40%), and 81.3 (60%)	-	↓ 42.3 (control), 39.6 (20%), 40.7 (40%), and 32.4 (60%)	[44c]
16	Engineered cementitious composite (ECC)	0.3	0, 5, 10, 30, 40, and 50% as replacement of fly ash	-	-	-	-	<u>@ 120 days</u> ↑ from 35 to 40 @10% and ↓ to 32 @ 50%	↑ from 3.19 to 3.6 @10% and ↓ to 1.8 @ 50%	-	[44q]

2.5 Application of RM in masonry bricks

Numerous studies have been published on the potential use of RM in the production of masonry brick. In 1987, Amritphale and Patel[42a] used RM in brick manufacturing and concluded that RM can be employed up to 50 wt.% in the production of building bricks. Atan et al. [42b] examined the impact of RM on the properties of fired clay bricks in order to create an eco-friendly porous ceramic brick. According to the findings, the potential use of RM decreases bulk density and compressive strength while increasing porosity, resulting in greater thermal insulation. The inclusion of 30 wt.% RM yields a brick with the lowest thermal conductivity (0.72 W/mK) and satisfactory compressive strength (32.4 MPa). Singh et al. [42c] examined the performance of bricks made with RM as a binder. It was found that RM-based brick has excellent mechanical characteristics and can be categorized as first-class brick. The optimum RM content was found to be 30 wt.%; however, bricks made with 50% RM also demonstrated an acceptable dry compressive strength of 8.7 MPa. Additionally, the presence of RM promotes geopolymerisation and increases compactness while increasing the number of shrinkage cracks.

Arroyo et al. [^{42d}] evaluated the performance of fired clay bricks with a high RM ratio as replacement for traditional clay. The study found that bricks fired at 1100 °C with 80 wt.% RM exhibit better mechanical properties, particularly a compressive strength greater than 16 MPa, which is believed to be the ideal amount of RM that can be used in the production of fired bricks without posing an environmental risk. Another important point to emphasize is that the radiological behavior of RM-containing construction materials should be thoroughly investigated because RM contains more naturally occurring radionuclides than other building materials.[42d] RM was also utilized as a fly ash substitute in the production

of fly ash bricks, as opposed to control bricks without RM. Results showed that RM replacement reduced early strengths and flowabilities by up to 30%. On the other hand, the strengths remained high enough after 7 and 28 days to be utilized as a structural binder when making bricks. It is recommended to use NaOH, up to 5% of the water content, to reduce the detrimental effect of RM on the initial strengths and flowability of the binder. This is because NaOH significantly increased the degree of reaction in the matrix, which encouraged the formation of more calcium silicate hydrate (C-S-H) and resulted in substantial refinement of the pore size at an early age.[42e]

He et al. [42f] reported that the best properties of RM-based fired bricks were obtained at an RM content of 40%, a sintering temperature of 1050 °C, and a sintering time at 2 h. Additionally, RM-based fired bricks made under ideal conditions had leaching toxicity and a radioactivity index that were below permissible limits and therefore safe for the environment. Similarly, Liu et al. [42g] demonstrated that the RM-based ceramic bricks when sintered at 1125 °C exhibit significant enhancements in physical and mechanical performance. Furthermore, the pH and conductivity of RM-based ceramic brick are significantly lower than those of raw RM leaching solution due to the stabilized aluminosilicates formed during the sintering process, preventing alkaline harm to the environment. Sudhir et al. [42h] developed fired bricks made of 100% RM and RM incorporated with fly ash and Ground Granulated Blast Furnace Slag (GGBFS). The results demonstrated that the compressive strength of the RM-based bricks increased as the fly ash and GGBS dosage increased, confirming that using RM to make bricks is a feasible option. Figure 16 displays the compressive strength of RM-based bricks from various studies at 28 days of curing, demonstrating that an acceptable strength of various bricks can be achieved when the RM content does

not exceed 40 wt.%. However, the higher the RM content, the greater the water absorption of the brick (Figure 17). Table 3 summarizes the properties of bricks made of composites containing fly ash, geopolymer, fired clay, etc., and RM.

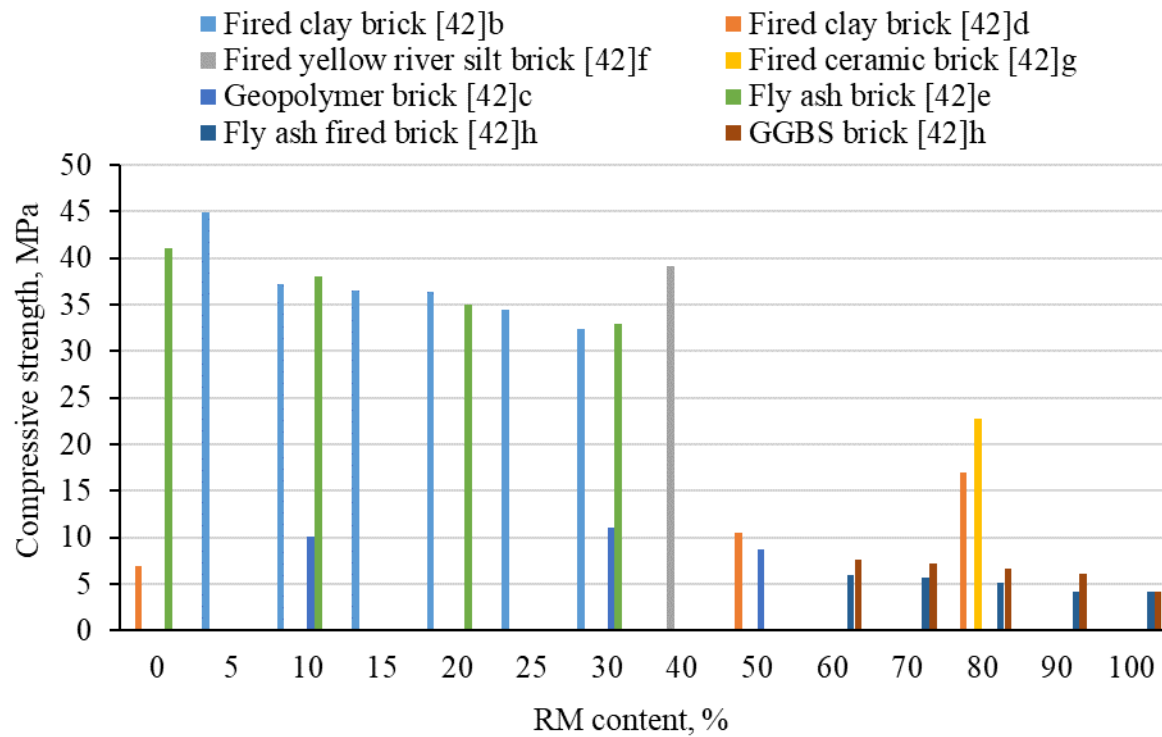


Figure 16 Compressive strength of RM-based bricks at 28 days.

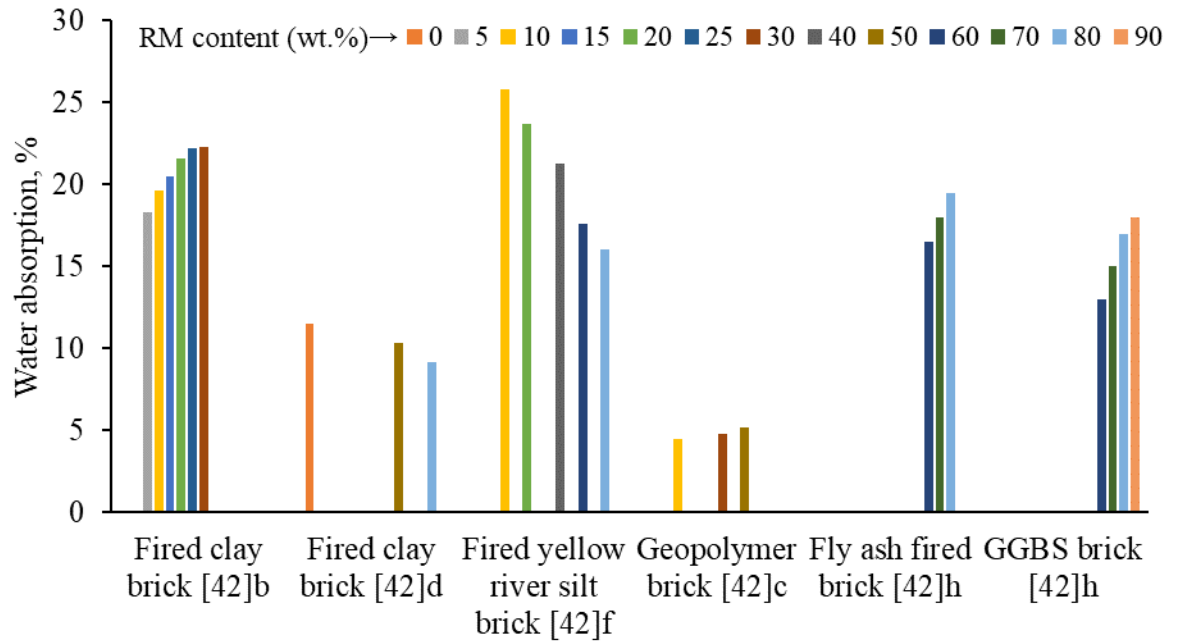


Figure 17 Water absorption of RM-based bricks at 28 days

Table 3 Properties of masonry bricks containing RM at 28 days curing age.

Type of building brick	Parameters	RM content (wt. %)	BD (kg/m ³)	LOI (%)	IRA (kg/m ² /min)	WA (%)	AP (%)	ThC (W/mK)	CS (MPa)	Ref.
Fired clay brick	Sintered Temp. 1000 °C	5	1799	8.5		18.3	33	0.78	44.9	[42b]
		10	1782	7.2		19.6	34.8	0.78	37.2	
		15	1770	7.4		20.5	36.3	0.77	36.5	
		20	1748	7.6		21.6	37.5	0.75	36.4	
		25	1739	7.8		22.2	38.5	0.73	34.5	
		30	1760	8.2		22.3	39.1	0.72	32.4	
Geopolymer brick		10	1884		1.09	4.49			10.1	[42c]
		30	1924		1.14	4.79			11.1	

	1:1 binder: aggregate ratio	50	1998		2.35	5.15			8.7	
Fired clay brick	Sintered Temp. 1100 °C	0	1830			11.5			6.9	[42d]
		50	1982			10.3			10.5	
		80	2150			9.2			17	
Fly ash brick	0.4 w/b ra- tio	0							41	[42e]
		10							38	
		20							35	
		30							33	
Fired Yellow River silt and RM brick	Sintered Temp. 1050 °C	10		5.7		25.8				[42f]
		20		6.3		23.7				
		40		8.2		21.3			39.1	
		60		9.8		17.6				
		80		11.4		16.0				
Fired ce- ramic brick	Sintered Temp. 1125 °C	80	1680				47.1		22.7	[42g]
RM+Fly ash fired brick	Sintered Temp. 700 °C to 900 °C	100	910			NA	32		4.2	[42h]
		90	1090			NA	32		4.2	
		80	1500			19.5	34		5.1	
		70	1800			18	36.4		5.7	
		60	1860			16.5	38.7		5.9	
RM+GGBS brick	Sintered Temp. 700 °C to 900 °C	100	910			NA	32		4.2	[42h]
		90	1600			18	35		6.1	
		80	1720			17	36.6		6.6	
		70	2020			15	38		7.2	
		60	2050			13	39		7.6	

2.6 Application of RM in Ceramic

Another significant use for RM is in the production of ceramic building materials, which is a major consumer of a variety of raw materials.[53] Among the numerous studies on the use of RM in ceramic production, Wang et al. [43a] studied the performance of ceramic tiles containing RM subjected to various sintering temperatures. The study discovered that substituting the raw material constituent with RM and increasing the sintering temperature could result in exceptional performance of ceramic floor tiles. Xu et al. [43c] examined the influence of adding various RM on the performance of fired ceramic tiles. It was found that the properties of fired ceramic tiles incorporating 40 wt.% RM were significantly improved, with up to 9.92% enhancement in water absorption and up to 60.8 % enhancement in flexural strength, but at lower sintering temperature. This is due to the fact that RM in its raw form have well-crystallized phases below 1100 °C, and low firing temperatures result in non-excessive liquid phases, which densify the matrix. In conclusion, RM-based ceramics are suitable for use as raw materials for ceramic tiles due to their superior properties, particularly their low water absorption and high flexural strength. Yang et al. [43b] developed two-layer ceramic tiles with a flexural strength of around 51 MPa using RM and bauxite tailings as the primary materials in the floor tile body. Babisk et al. [54] examined the feasibility of manufacturing roofing ceramic tiles for building construction from plain RM and clay. The results revealed that RM, particularly in the proportion of 50 wt.% at 1050 °C sintering temperature, has sufficient properties to be utilized as a raw material in the clay ceramic industry for roofing tiles. Similar findings were obtained by Pérez-Villarejo et al., [55] who studied the influence of adding RM into ceramic matrix and found

that the optimum inclusion of RM was 50 wt.%. This is because RM generates a sizable amount of vitreous phase, which significantly modifies and improves the mechanical and physical characteristics of ceramic products.

Contreras et al. [56] developed an environment-friendly ceramic tiles using RM from ilmenite. Contemporary ceramic tiles are more technologically advanced and have lower impact on the environment than conventional ones. The use of RM as an additive during the sintering processes increased flexural strength by up to 15% and reduced apparent porosity and water absorption by up to 50%. He, et al. [57] studied the influence of RM content on the performance of ceramic tiles and obtained the main phase of samples of hematite, feldspar, gehlenite, and nepheline with compressive strengths of 40 MPa. The ideal firing conditions were 1050 °C, 2 h, and 20 wt.% RM content. A significant portion of the high compressive strength of ceramic tiles is due to the glass phase, which is formed by silicon dioxide and alkali and alkaline earth oxides. However, there are some significant challenges associated with their use as ceramic building materials, such as low density and ensuring that ceramic bodies have adequate compressive strength. Table 4 summarizes the properties of RM-based ceramic tiles based on the findings of previous studies.

Table 4 Summary of the properties of RM-based ceramic tiles based on the existing studies.

Sintering temp. (°C)	RM content (wt. %)	BD (kg/m ³)	WA (%)	AP (%)	FS (MPa)	CS (MPa)	Ref.
(1150–1180) for 2 h	65 – 70	Reduced from	Decreased from 9.3 to 5.5	Decreased from 28 to 19	Reduced from	NA	[43a]

		1820 to 1450			142.3 to 185.6		
930 – 1000	40	2220 @ 930 °C			Increased from 43 to 51	NA	[43b]
940 – 1140	40	Ranged between 2650 - 1680	The max. ranged between 4.93 – 0.8	Ranged between 34.5 – 1.1	The max. ranged between 61- 48.7	NA	[43c]
850, 950 and 1050	33.3, 50, and 100	De- creased from 2333 @ 0% RM to 1696 for 100% RM	At 100% RM, WA is 27% and at 0%RM WA is 21% @ 1050 °C	NA	NA	At 100% RM, CS is 8.1 MPa and at 0% RM CS is 2.3 MPa @ 1050 °C	[54]
Up to 950 for 1 h	0 - 100	1760 at 0% RM and de- creased to 1720 at 20% RM then increased to 1802 at 100% RM.	20.9 @ 50% RM and 15.1% at 100% RM	NA	NA	45 at 0% RM and decreased to 13.9 at 20% RM then in- creased to 53 at 50% RM.	[55]
1150	3, 5, 7, 10, 30 and 50	de- creased from 2360 to 2270	Increased from 5.3 to 9.18	Ranged between 12.5 to 20.9	De- creased from 35.1 to 30.8	NA	[56]

1000 - 1100 for 2 h	20	Ranged between 1590 to 2150	Decreased from 24.1 to 4.38	NA	NA	Increased from 28.3 to 36.5 at 1050 °C	[57]
Note: wt. %=weight percentage; RM=red mud; BD=bulk density; WA=water absorption; AP=apparent porosity; FS=flexural strength; CS=compressive strength; NA=Not Available.							

2.7 Application of RM in Geopolymer

Due to the fact that RM contains aluminosilicates, a recent method for its recycling can be found in the development of a geopolymer.[58] A geopolymer is an alternative binding material that can replace OPC by up to 100%, making it an environmentally friendly green material. The factors influencing the mechanical characteristics and microstructure of geopolymers incorporating RM and other waste materials, such as granite waste powder etc., have been thoroughly reviewed.[48b] Additionally, the sustainable use of RM, fly ash, and slag in the production of geopolymer composites[30a, 41a] and alkali activated concrete [48f] has been addressed.

The compressive strength of the composite increased to 30 MPa at 5 wt.% RM inclusions in the geopolymer matrix; however, as the RM content increased, the strength decreased.[59] On the other hand, a geopolymer with 20 wt.% RM and 80 wt.% fly ash exhibited a compressive strength of 20 MPa.[48g] Various researchers have reported comparable results,[48h, 48i], where the addition of 50 wt.% RM can produce a geopolymer with a compressive strength between 15 and 20 MPa. Aygörmez[48a] utilized RM as a binder to produce reinforced metazeolite-based geopolymer composites. It was concluded that the

addition of 25 wt.% of RM was the optimal content to produce the highest performance for geopolymer composites. Furthermore, as the RM content increased, brittle fracture formation was observed.

The ductility of the composite with RM was enhanced due to the fine particle size and high specific surface area of RM. At higher rates of RM usage (>25 wt.%), the enhanced ductility began to deteriorate. Because RM contains hematite, a highly absorbent mineral, it can be used in geopolymers to help reduce the amount of heavy metals, toxic chemicals, and radioactive waste.[60] Mudgal et al. [25] examined the performance of fly ash with RM geopolymer and concluded that RM enhanced the geopolymerization process and mechanical properties of the geopolymer composite. The compressive strength of fly ash-based-RM geopolymer was 65 MPa after 28 days of curing, whereas it was around 53 MPa for fly ash-based-geopolymer without RM. Furthermore, the inclusion of RM increased the Si/Al and Na/Al molar ratios compared to fly ash geopolymer, which contributed to the enhancement in the geopolymerization reaction, as evidenced by SEM analyses, which indicate a more dense microstructure due to its filler effect. Ge et al. [48c] studied the microstructure and mechanical properties of fly ash geopolymer-incorporated RM, including the effect on curing, temperature, and RM to fly ash ratio. According to the findings, stronger fly ash-RM geopolymer pastes require longer curing duration and higher curing temperatures, whereas shorter curing times and lower RM to fly ash ratios result in a reduction of mechanical properties. Additionally, when the microstructure and mechanical performance are taken into account, a RM to fly ash ratio of 39% is the critical upper limit for producing fly ash-RM-based geopolymers under ambient curing conditions. Sun et al. [48d] studied the mechanical and environmental properties of an RM-based geopolymer with ground

granulated blast slag (GGBS) and metakaolin (MK). It has been reported that as the RM content increases, the mechanical behavior of the resulting RM-based geopolymer decreases. Furthermore, when compared to the results of RM-based geopolymer incorporated GGBS and MK with a similar RM content, the MK geopolymers exhibit better mechanical properties, which could be attributed to the higher reactivity, increased fineness, and layered structure of MK. The pH values fluctuated slightly but stayed between 11 and 12 throughout the experiment. Alam et al. [61] examined the mechanical properties and durability aspects of a GGBS-based geopolymer containing RM, concluding that the RM-GGBS geopolymer composite could be utilized as a geomaterial. Yang et al. [48g] produced geopolymer from RM slurry and class-F fly ash. The outcomes showed that the RM-fly ash geopolymer composite had a compressive strength greater than 17 MPa, which is considered sufficient for use in structural cement. RM-geopolymer had a compressive strength of 15.2 MPa after curing at ambient temperature with a significantly lower NaOH concentration due to the high alkalinity of RM, which aided in geopolymerization, but more NaOH was required to achieve the highest compressive strength. In contrast, both fly ash and RM geopolymers were unable to reach usable strength when activated with only the NaOH solution, a problem that was solved by curing at elevated temperatures or using a composite activator.^[48h] A novel geopolymer composite was also developed using RM and rice husk ash (RHA). Long-term curing greatly improves Young's modulus and compressive strength, whereas ductility decreases. Higher RHA/RM ratios produce stronger and more ductile materials; however, excessive RHA concentrations can have the opposite effect. The compressive strengths of the synthesized geopolymers ranged from 3.2 to 20.5 MPa, with nominal Si/Al ratios of 1.68-3.35.^[48i]

The $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio, $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ ratio, and water/binder ratio of the geopolymer composition all had an impact on the setting time of the RM-based geopolymer. When the $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio reaches 4.0, the initial and final setting times tend to increase, resulting in a slight decrease in setting times. The setting time increased due to the silica-silica reaction, which occurs as a result of the increased silica content and is a slower process than the silica-aluminum reaction.[62] Additionally, the increased alkalinity of RM caused a decrease in compressive strength, inadequate workability, and rapid setting.[48b] It was discovered that adding RM significantly decreased the flowability of the RM-fly ash geopolymer mixes by more than 20%. However, the slump flow increased by about 17% when the water to binder ratio was raised from 0.40 to 0.48. The flowability of the mixes improved with the inclusion of NaOH at higher concentrations of 1%, 3%, and 5% due to the dispersing effect of NaOH.[42e]. In general, the inclusion of NaOH was reported to be more advantageous than increasing the water content in RM-based geopolymers to achieve sufficient flowability and strength. Figure 18 and Figure 19 depicts the flexural and compressive strengths of geopolymer-incorporated RM. The mechanical strength of calcined zeolite geopolymer, RHA-based geopolymer, GGBS-based geopolymer, and MK-based geopolymer increased with increasing the RM content up to 50 wt.%, whereas fly ash-based geopolymer decreased significantly with increasing the RM content up to 50%. Table 5 summarizes the geopolymer properties found in the existing literature.

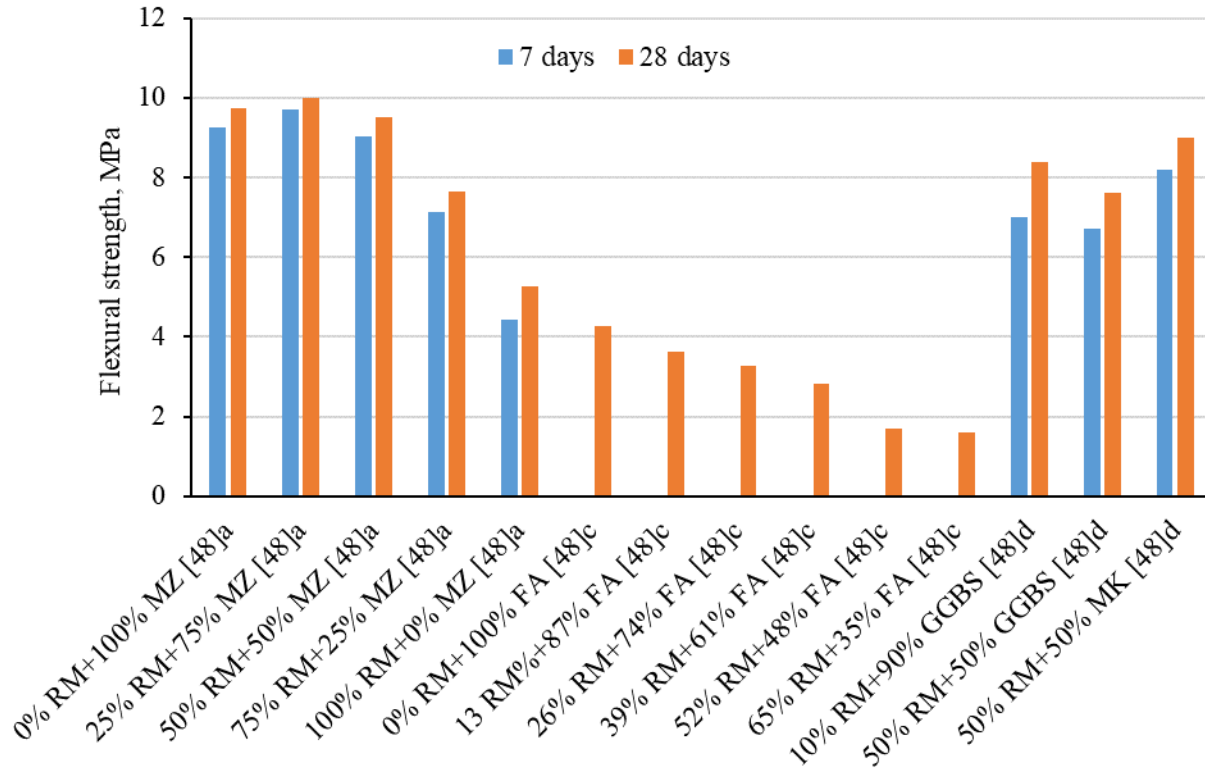


Figure 18 Flexural strength of RM-based geopolymer.

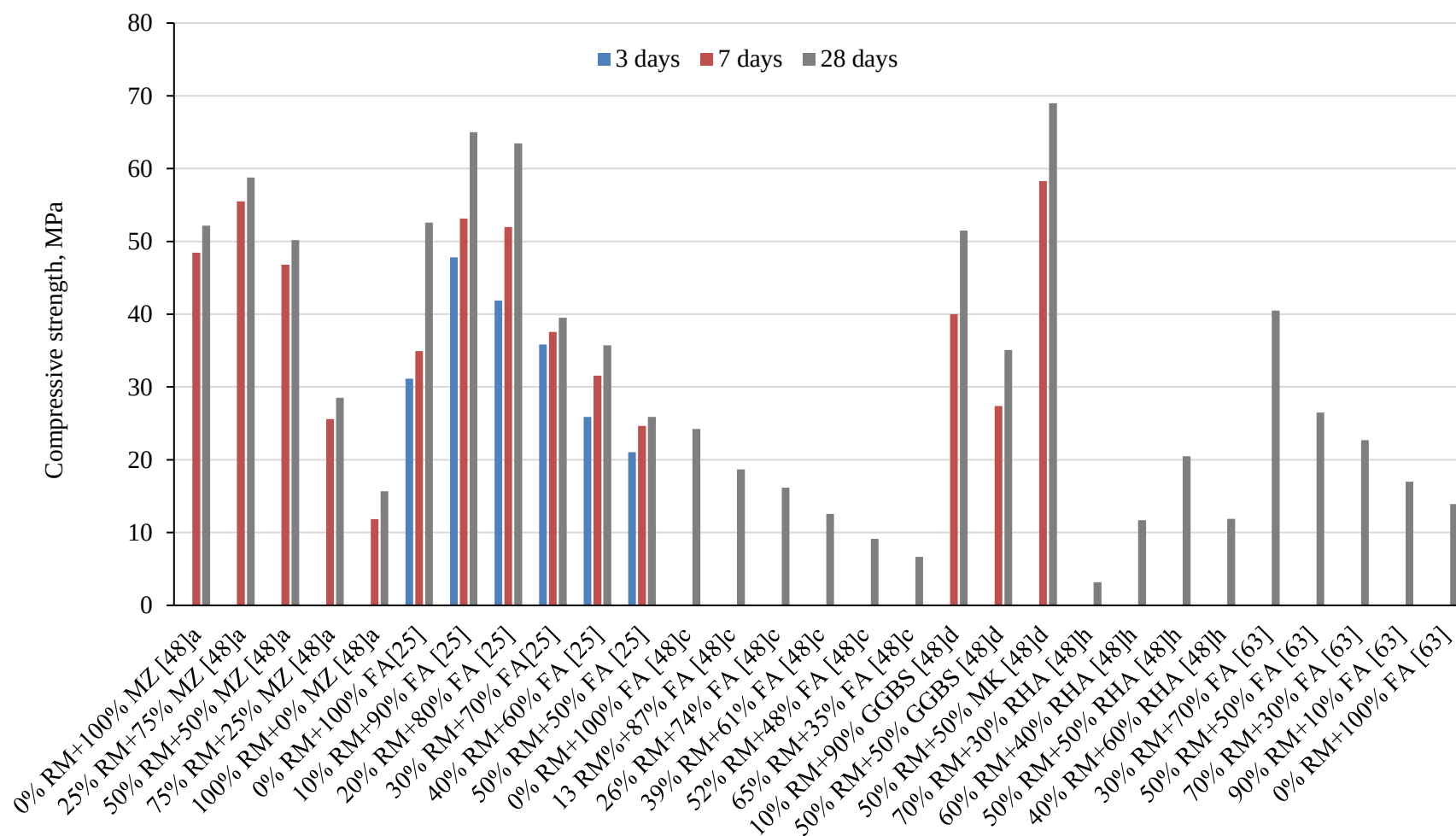


Figure 19 Compressive strength of RM-based geopolymer.

Table 5 Summary of the geopolymer properties obtained from existing literature at the optimum RM content.

Geopolymer	RM content (wt. %)	Optimum RM content (wt. %)	Curing age, (days)	Setting time		AP (%)	FS (MPa)	CS (MPa)	Ref.
				Initial	Final				
MZ-based geopolymer	25, 50, 75, and 100	25	7				9.7	55.5	[48a]
			28				10	58.9	
			56				9.8	57	
Fly ash geopolymer	10 – 50	10	3					47.8	[25]
			7					53.2	
			14					58.8	
			28					65.8	
Fly ash geopolymer	0 - 78	39	28	↑from 119.6 @ 0%RM to 220.3 @ 39% RM	↑from 274.8 @ 0%RM to 367.1 @ 39% RM	↑ from 0.191@ 0% RM to 0.268 @ 39% RM	↓ from 4.3@ 0% RM to 2.8 @ 39% RM	↓ from 24.2@ 0% RM to 12.6 @ 39% RM	[48c]

GGBS-based geopolymer	10 and 50	50	7				6.7	27.4	[48d]
			28				7.6	35.1	
MK-based geopolymer	50	50	7				8.2	58.3	[48d]
			28				9	69	
RHA-based geopolymer	70, 60, 50, and 40	50	28					Ranged between 3.2 to 20.5	[48i]
Fly ash geopolymer	0, 90, 70, 50, 30	30	28 days @ 60 °C and 6% NaOH (M)					13.9, 17, 22.7, 26.5, 40.5	[63]

2.8 Applications of RM in Catalysis

2.8.1 RM as Hydrogenation Catalysts for Value-Added Chemicals

Unsaturated organic compounds can be transformed into a series of important building blocks by using either direct or indirect addition of H^2 . These transformations often require the activation of the hydrogen molecule, if it is molecular hydrogen, by suitable catalysts. In this case, the composition of RM may be ideal for use as a catalyst in the hydrogenation reaction. Therefore, a series of RM-based catalysts are used in the hydrogenation reactions of coal,[64] biomass,[65] oil shale,[66] and petroleum residue.[67]. A variety of iron oxide catalysts, such as RM, iron ore, pyrites etc., have been successfully used in the coal hydrogenation reaction. The results showed that RM-based catalysts performed well by resisting the catalyst poisons like sulfur found in the coal.[68] However, the overall efficiency of the catalyst is rather low. This is mainly due to the low surface area and high agglomeration of the RM-based catalyst. Therefore, size reduction, higher dispersion, and higher surface area may be crucial in modifying the RM to achieve the optimum results in terms of conversion and selectivity.

Global warming and ocean acidification caused by greenhouse gases have become major concerns in the 21st century. The conversion of carbon dioxide, which is a non-toxic, readily available, and abundant carbon resource, from the environment into valuable products could be a significant step forward. In addition, as fossil fuel reserves deplete, CO_2 conversion as an alternative carbon feedstock for the production of commodity chemicals and fuels becomes increasingly important. Catalytic CO_2 hydrogenation has recently mainly focused on the production of CO ,[69] CH_4 ,[70] CH_3OH ,[71] C_2 - C_4 olefins,[72] gasoline,[73] aromatics.[74] However, to convert the chemically inert CO_2 requires extreme

reaction conditions. As a result, numerous Fe-, Co-, Ni-, Cu-based catalysts immobilized on various supports, such as zeolite, perovskite, silica, and activated carbon, have been developed and successfully applied for CO₂ hydrogenation. However, the RM for hydrogenation reactions is rarely investigated. Recently, Artem et al.[75] reported a CO₂ hydrogenation reaction using potassium-promoted RM-based catalysts (Fig. 20) for the selective production of lower olefins, and they achieved 45% conversion with 36% lower olefins (C₂-C₄) selectivity at 375 °C under 30 bar of CO₂:H₂ (3:1) with a flow rate of 9600 mL.g⁻¹.h⁻¹. Unpromoted RM, on the other hand, is capable of hydrogenating chemically inert CO₂, especially at higher temperatures (375 °C), with 42% conversion and 15% lower olefins selectivity. The stability of the K-promoted RM under very high pressure remains longer (50 h). Also, the reactivity of the catalyst K-promoted RM was compared with the commercially available iron oxide, and the results showed that both catalysts display similar performance and even slightly better than that of the commercial counterpart.

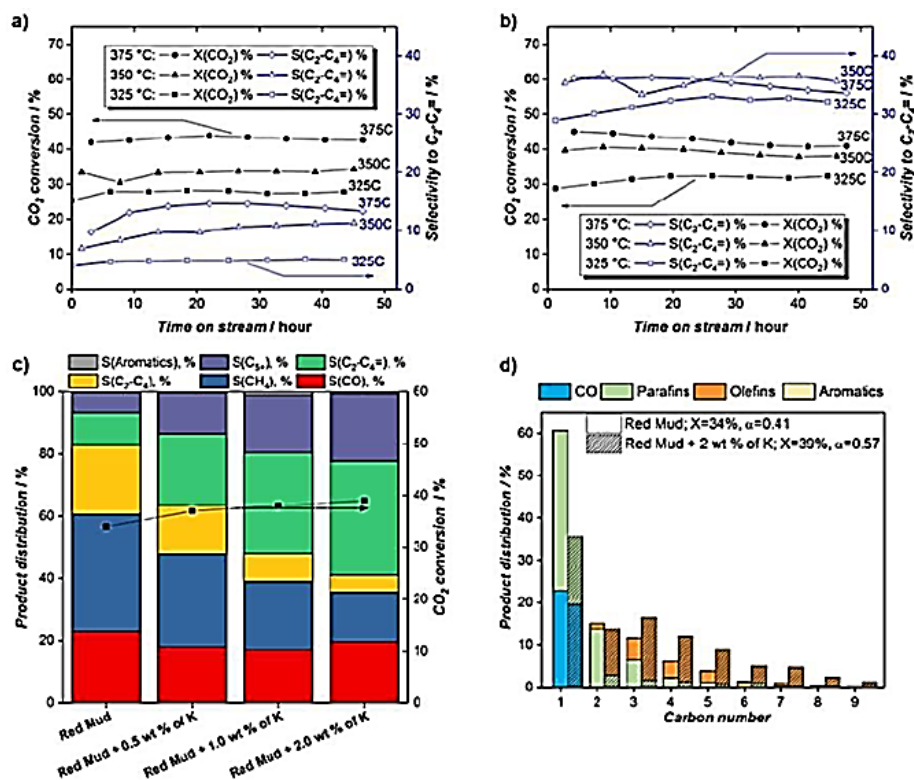


Figure 20 Catalytic results of RM and K-promoted RM in the hydrogenation of CO₂. a) CO₂ conversion and selectivity to C₂-C₄ olefins at 325 °C, 350 °C and 375 °C for RM. b) CO₂ conversion and selectivity to C₂-C₄ olefins at 325 °C, 350 °C and 375 °C for RM promote

2.8.2 RM as Hydrodechlorination Catalysts

Chlorinated organic compounds, such as alkanes, olefins, and other chlorine-containing compounds, are critical raw materials and organic solvents in the pharmaceutical, plastics, polymer leather, electronics, and pesticides industries. However, the by-product generated during the production of the chlorinated organic moieties poses a serious threat to the environment and human health. Most of these wastes have been accumulating for a long time and are difficult to degrade over time. As a result, chemists are very interested in the degradation of these toxic organic compounds. In this context, a series of transition-metal-based hydrodechlorination catalysts have been developed to degrade the chlorophenol.[76] Noble metals, such as Pd, Rh, and Pt, have been used successfully as dichlorination catalysts.[77] However, the strong affinity of the produced chloride for a noble metal quickly

deactivates the catalyst. In addition, such adsorption promotes agglomeration and carbon deposition at Pd sites. As an alternative to noble metals, base metal-mediated catalytic systems are more robust, less expensive, and more recyclable. In this context, numerous earth-abundant metal sulfides of V, Cr, Mn, Fe, Co, Ni, and Mo are used for the hydrodechlorination reaction. However, these are highly susceptible to being poisoned by the produced chloride.

In these circumstances, RM has been explored as a catalyst for the tetrachloroethylene hydrochlorination reaction and detoxification of C2-chlorohydrocarbon.[49,78] The results showed that sulfided RM is an active catalyst due to the higher iron sulfide content. Ordóñez et al. [78] explained that sulfidation of the catalyst, higher operating temperature, and enhanced pressure had all positively influenced the catalytic activity of the RM.

2.8.3 H RM as Hydrocarbon Oxidation Catalysts

RM is at the forefront in the oxidation of volatile organic compounds (VOCs), a highly toxic environmental pollutant, to a variety of valuable fine chemicals. For example, toluene has been successfully converted to its corresponding oxygenates by using RM as a catalyst. Lemonier et al.^[79] reported that heat-treated RM in an air stream at 500 °C produces predominantly Al₂O₃ and Fe₂O₃, resulting in efficient toluene oxidation. The higher reactivity of calcined RM is correlated to the higher degree of Fe₂O₃ formation from pure RM. It is also demonstrated that the reaction rate of RM is comparable to that of the commercially available pure Fe₂O₃ in the toluene oxidation reaction. Paredes et al.[80] reported a methane combustion study in which the iron was extracted from the RM using the dissolution-precipitation method, followed by two hours of calcination at 500 °C in the air. The cata-

lytic results for methane combustion under atmospheric pressure at two different temperatures, 300 °C and 626 °C, were compared to those of the commercial catalyst Cu-Cr-Ti. The RM activated using the Pratt and Christoverson method exhibits a higher catalytic activity and enhanced thermal stability.

2.8.4 Catalyst Towards Prevalent Air Pollutants Remov

Recently, RM-based catalysts have been used to remove prevalent air pollutants[81] However, the acid sites on the surface of the catalyst can be consumed by the alkaline components in RM, altering the structure of the catalyst and reducing NH_3 adsorption.^[82] Therefore, RM could be activated primarily by eliminating alkaline groups like Na_2O and CaO , which results in an improved specific surface area and more advanced pore structure. An alkaline metal can be eliminated by acid washing and calcination can change the goethite structure in RM to a consistent Fe_2O_3 structure and cause some salts to decompose. Using this treatment method, the RM was activated and displayed high de NO_x activity between 300 and 400 °C.[83] Wang et al. [84] used acid washing and calcining to create a series of RM catalysts with several other binders. Sulfuric acid and RM were combined with hydrothermal treatment to create an RM-based catalyst, which was then pH adjusted with ammonia. In addition to having a high tolerance for water and SO_2 , the RM catalyst demonstrated high catalytic activity by converting over 80% of NO_x at temperatures between 350 and 450 °C.[85] It was discovered that the RM catalyst had a highly dispersed Fe active site population, a large specific surface area, and an amorphous structure. These characteristics made the catalyst ideal for NH_3 adsorption, an essential step in the NH_3 -SCR of NO_x . [86] Additionally, RM can be pretreated with sulfuric acid, nitric acid, and hydrochloric acid.[87]

At 150 °C, a catalyst made of RM and fly ash with Co at 5 wt.% and Mn/ZSM-5 at 10 wt.% can achieve 98.7% NO_x conversion and 90.6% N₂ selectivity. The catalyst was also highly resistant to water and sulfur.[88] Chen et al. [89] created a monolithic honeycomb RM catalyst using extrusion molding and HCl leaching for RM treatment. At 350°C, the mixture containing 3% activated carbon demonstrated the highest catalytic activity, with NO_x conversion exceeding 90%. Van Nguyen et al. [90] used a hydrothermal method to create the mitochondrial nuclear retrograde regulator 1 (MnRR1) catalyst from RM-rice husk ash. The highest CO catalytic oxidation activity was shown by 3.0 MnRR1, which had a 3 wt.% Mn loading and 100% CO conversion at 280°C, as shown in Fig. 21.

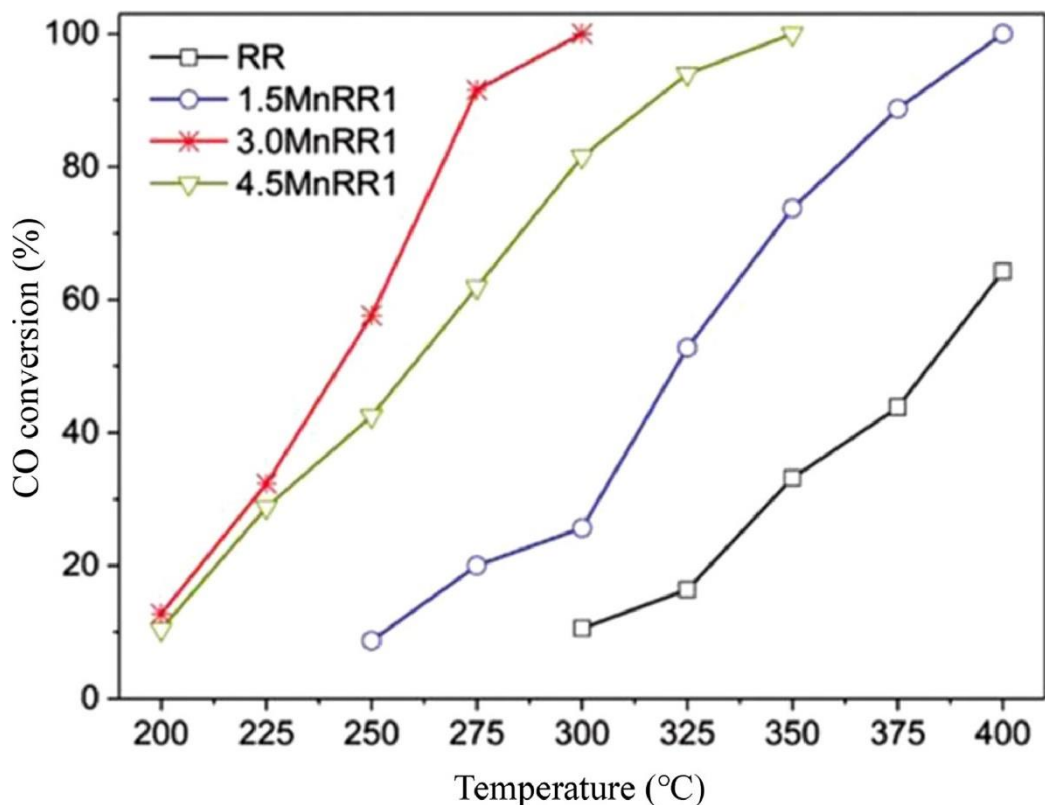


Figure 21 Catalytic activity of RM-rise husk ash catalyst. Adapted with permission,[90] Copyright 2020, RSC.

2.9 Conclusion

The following conclusions can be drawn from the extensive literature covered in this review:

- 1- Several potential applications for red mud (RM), an industrial waste material generated as a byproduct of aluminum production, have been investigated. RM can be used in, for example, construction and building materials, bricks, ceramics, catalyst, environmental wastewater treatment, metal recovery, etc.
- 2- Morphological, elemental, and structural characterization studies on RM indicate that it is a porous and fine particle matter with particle sizes in the micron range. It

is an amorphous material with a high concentration of oxides derived from its bauxite source ore, such as iron, aluminum, titanium, etc.

- 3- RM has the potential to pollute the air, soil, water, and ecosystem, if not properly disposed of or treated.
- 4- Among the different applications, utilization in the construction industry is one of the most promising, large-scale applications of RM, owing to the significant amount generated when compared to recycling.
- 5- Because of its fine particle size, RM incorporation in cement composites densifies the microstructure and thus improves the mechanical and durability performance of the cement composites. However, due to its mineralogical composition and high alkalinity, it degrades the performance of cement composites at high concentrations (greater than 10%). Although RM decreases workability because of higher water requirements attributed to its finer particle size, this can be compensated for in cement composites by using suitable superplasticizers. It also demonstrates improved performance in blended cement composites with fly ash, GGBS, and other waste materials by facilitating pozzolanic reactivity thus contributing to concrete strength.
- 6- Due to the presence of aluminosilicate in its composition, RM is also beneficial as a component in geopolymer composites, contributing to the geopolymerization reaction and enhancing the performance of the geopolymer concrete. Thus, another alternative and more sustainable technique for its disposal.

- 7- The RM has been utilized as a catalyst or catalyst support for various reactions, such as hydrogenation, hydrodechlorination, hydrocarbon oxidation, and air pollutants removal.
- 8- Because RM is radioactive, it is not advisable from a radiation safety perspective to recycle RM at a high rate (80%) when making bricks. RM can be used as an active catalyst to reduce air pollution, which not only makes use of solid waste but also lowers catalyst costs.
- 9- Although RM has been used in a number of potential applications, its consumption is still quite low.
- 10- The information presented in this review would aid in the development of a clear road map for utilizing RM waste to mitigate environmental concerns while increasing economic benefits, either by utilizing RM in construction and building materials or as a catalyst and catalyst-support material.

2.10 Future Research

Despite of a small amount of research on the utilization of RM in diverse applications, it is still necessary to advance other value-added products with this material.

This review lays the groundwork for future research into utilizing the RM as a building material in the production of cementitious composites, concrete, masonry bricks, and other materials that can benefit the construction industry. However, further research is required to explore the use of RM in other civil engineering applications, such as asphalt concrete, environmental wastewater treatment, electrochemical energy storage for structural supercapacitors [91] 3D concrete printing, etc.

Although some properties of concrete have been explored utilizing RM, more research is needed to study its effect on fresh, rheological, mechanical, and durability characteristics of concrete, such as rate of hydration, plastic viscosity, yield strength, bond strength, carbonation, reinforcement corrosion, fire resistivity, etc.

Its application as a component in composite/anticorrosive coatings for preventing steel corrosion[92] and enhancing concrete structure durability[93] can be explored.

Following this review, the research trajectories of utilizing the RM as a catalyst and catalyst-support material are expected to accelerate.

RM can be used as a constituent in various cementitious composites containing recycled waste materials, enhancing their performance, and contributing more towards sustainability.

More research is needed in managing bauxite residue waste streams from alumina plants.

CHAPTER 3

EXPERIMENTAL PROGRAM

The most basic capacitor consists of two conducting plates submerged in an electrolyte and an insulating surface between them. These plates possess different charges; one is negative and the other is positive. The former acquires positive charges from the electrolyte when a voltage is applied across the capacitor, whereas the positive plate acquires negative ions. The separation and the charges in the capacitor produce an electric field, which charges the capacitor. This is because the insulating material prohibits the movement of electrons. When necessary, the two plates may instantly deliver this pair of charges after maintaining them for a lengthy period. Supercapacitors are simple capacitors with incredibly enormous charge storage capacities. The idea of supercapacitors becomes more relevant when considering the global rise in energy demand, the quick depletion of nonrenewable energy supplies, and environmental issues like climate change and global warming. For these reasons, the exploitation of unconventional renewable energy sources is of great interest on a global scale. Because of its high power density, high specific capacitance, and improved cycle stability, supercapacitors stand out among other energy storage technologies as highly effective portable power storage units that may be used for a variety of applications, including consumer electronics and electric vehicles.

Batteries and supercapacitors have fundamentally different energy storage mechanisms and electrode compositions. Supercapacitors' distinctive performance, which distinguishes them from batteries, should thus be preserved. For example, some of the characteristics

that distinct a supercapacitor are the following: power density, cyclability, charge/discharge ability, self-discharging ability, and finally, safety and cost. Table 1 shows a comparison between conventional batteries and supercapacitors. As the table suggests, the typical range of power and energy densities is around 4-5 Wh/kg and 10–20 kW/kg, respectively. However, recent research work indicates that the aforementioned values can be surpassed in the near future due to the quick development in this technology. Nonetheless, for now, supercapacitors have lower energy density compared to batteries, but they have other advantages. The most notable one is that supercapacitors usually have a practically limitless lifespan. In addition, they provide a larger temperature range, faster charging speeds (compared to battery materials), improved safety, increased dependability, and maintenance-free operation. Furthermore, supercapacitors usually provide pulse energy. That is, they can provide a high amount of energy in a short time i.e. they have fast discharge time, which can typically be less than one second. Nonetheless, supercapacitors are becoming in demand for usage in conventional applications which do not require such a fast discharge time as well. This includes applications in automotive industry, particularly public transportation vehicles like buses and tramways, as a primary and supplementary energy source. Supercapacitors can store charge during regenerative energy braking and are particularly helpful for starting motors. They also have uses in electronics, household appliances, industrial machinery, and renewable energy.

Researchers' interest has recently been drawn to structural supercapacitors because they can store electrochemical energy in addition to supporting mechanical strain. As a result, research into the basic and practical features of structured supercapacitors has been conducted on a global scale. Additionally, because they are readily available and reasonably

priced, the potential for employing building materials for transdisciplinary applications is being researched. As a result, materials used in building might be thought of as prospective candidates for structural supercapacitors.

One of the building materials that are thought to be a good candidate for supercapacitor manufacturing is bauxite residue or red mud (RM). RM is a byproduct of the Bayer process, which is used in alumina production. This waste material is special because it contains iron oxide, thus the red color, and many other oxides. The Bayer method is used for over 95% of the world's alumina production and this method produces about 1 – 1.5 tons of red mud for every ton of alumina. There may also be a variety of additional components at low concentrations; these are always found as metallic oxides, such as nickel, copper, lead, chromium, manganese, and potassium. It may contain non-metallic components including carbon, and sulfur as well. Furthermore, a wide range of organic compounds, including alcohols, sugars, sodium, and oxalic acids can also be found in bauxite. The presence of these compound might be due to the use of crystal growth modifiers or flocculants. Table 1 shows the chemical composition of RM. Aluminosilicate, a compound contains silicon oxide and aluminum oxide, is also present in RM, which makes it useful as a component in geopolymer composites. It also contributes to the geopolymerization reaction and improves the functionality of geopolymer products such as geopolymer concrete. Hence, a different, environmentally friendly method of disposal. Physical aspects like particle size distribution and moisture content are additional essential considerations when evaluating the usage of RM to sustain straining actions. The range of particle sizes of bauxite leftovers varies from an alumina facility to another, but it is usually within the range of 1 mm in to less-than-one-micron. The median particle size, however, is typically in the 5–10 m range.

Some alumina manufacturing plants segregate the various sizes of RM particles, which facilitate the process of recycling, but some others don't.

Surprisingly, despite comprising of hematite (Fe_2O_3) mostly, a material that has been widely used in supercapacitor electrodes, red mud's potential as a pseudocapacitive material has not yet been thoroughly investigated. Galvanostatic Charge Discharge (GCD)

Galvanostatic Charge Discharge (GCD) tests (also called Constant Current Charging/Discharging) are frequently used to assess energy storage materials and systems, such as those found in electrochemical capacitors (ECs). In GCD, a material or system is charged and discharged within a predetermined potential limit by applying constant positive and negative currents; this process is frequently repeated for a number of cycles. In addition to determining the potential existence of irreversible Faradaic processes and assessing the quality of capacitive response, GCD profiles are also used to calculate various important electrochemical capacitors Figures of merit, including capacitance, capacity, energy, and power. It's important to mention that, here in this graphs, we meant the current not the current density. In the Figures below 37 and 38 which shows the GCD results from 0 to 1800 seconds and from -1 to 2500 seconds respectively, it can be seen that the least current value which is 0.0004 A gave us the highest supercapacitor capacity. While the lowest supercapacitor capacity recorded with the highest current value which is 0.003 A. Comparing both graphs, the graph from -1 to 2500 gave us more accurate and better results than the graph from zero to 1800 due to its excellent discharge curve and better intermediate resistance drop (IR drop).

3.1 Materials

The jute plants used in this work were harvested from Mominpur, Keshabpur, Jessore, Bangladesh and went under a retting process to yield jute sticks. Sodium bicarbonate (NaHCO_3) and hydrochloric acid (HCl) were obtained from Sigma-Aldrich. High purity nitrogen (N_2) gas was obtained from Specialty Gases Company Limited, Jubail, Kingdom of Saudi Arabia. The RM was obtained from Ma'aden Company (Al Jubail, KSA) and later ground into fine particles. To ensure the uniformity of the prepared RM, particle size distribution was carried out. Ordinary Portland cement (Type I) from Saudi Cement Company was used as a binder. Drinking water was used to mix the mortar at a 0.45 w/c ratio and, to further increase the workability of the mix, superplasticizer (Glenium) was added. Potassium hydroxide was also added. A typical mortar mix design was adopted, as shown in Table 6.

Table 6 mix design

Mix design for this experiment	
w/c	0.45
Cement (Type I)	335 (g)
Sand	685 (g)
S.P. (Glenium)	2% (by weight of binder)
RM% used	0%, 20%, 40%, 60%, 80%
KOH (Potassium hydroxide)	0.1 (g)

3.2 Preparation of jute sticks derived activated carbon (JC)

JC were prepared in accordance with [41]. In briefly, the preparation process started by cutting the jute sticks into small pieces then washing them with deionized (DI) water. After that, the sticks were dried at 100° C for a full day in an electric oven before they were ground to powder in a household blender. The powdered sticks were sieved in a 100 μm mesh sieve and only the passing particles were collected to be used through the experiment.

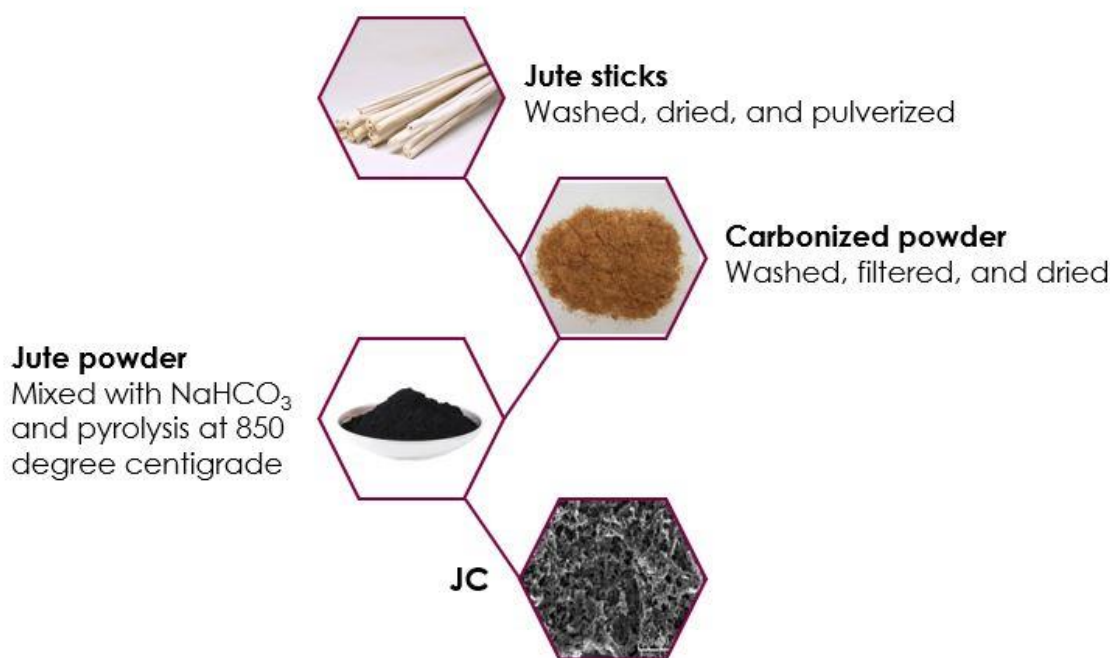


Figure 22 A schematic diagram showing the process of preparing jute sticks derived activated carbon (JC).

The obtained fine powder was mixed and homogenized with NaHCO_3 in a 1:4 ratio. The homogeneous mixture was then pyrolyzed at 850° C for 5 h in under an N_2 environment inside a tube furnace with a heating and cooling rate of 10° C/min and 5° C/min respectively. The mixture was then cooled in at room temperature and the carbonized product

was washed thoroughly with 0.5 HCl twice and finally with DI water to remove any impurity. The final product was then dried at 60° C for half a day in an electric oven. A schematic diagram for this process is shown in Figure 1.

3.3 Preparation of mortar samples

To conduct mechanical tests and durability tests, 50mm cubes, 25*25*285mm prisms, as well as 200*100mm cylinders at different RM% (ranging from 0% to 80% in increments of 20%) were prepared. The samples were submerged for about 7, 14, and 28 days. The remaining hardened mortar fragments from the mechanical tests were used for SEM analysis, while the raw powder was used to conduct XRD and XRF characterization tests. Figure 2 summarizes the civil engineering aspect of this experimental program.

3.4 Fabrication of electrodes and supercapacitor

The supercapacitor was prepared in three primary stages. The first stage is the preparation of activated carbon. The second stage is the electrode preparation. This process begins with cutting steel mesh into dimensions of 1 x 6cm. The electrodes are then cleaned using an ethanol bath for 60 minutes in a sonicator. This is followed by two rounds of rinsing with deionized water and subsequent drying. The third stage is the preparation of the electrode coating. The coating mixture is prepared with a ratio of 75:15:10 (Activated Carbon: Carbon Black: PVDF), using NMP as the solvent. This mixture is then applied onto the steel mesh and allowed to dry overnight at 80°C. To determine the mass of the activated carbon used, the electrode is weighed before and after coating. The difference in weight indicates the mass of the activated carbon. The area of the activated carbon electrodes was 3 cm² each. In the final stage, the supercapacitor is assembled by placing the electrode into the mold in a parallel arrangement. It was ensured that the distance between the two electrodes

of the supercapacitor was maintained at 3 mm while poring the mortar mixture. The sample was prepared by placing parallel steel meshes (electrodes) in a special mold and then pouring the RM mixture. The interior walls of the mold were coated with a thin oil layer before the casting. Care was taken to avoid coating the electrodes with oil.

3.5 Characterizations

The purity and mesostructured of the RM, OPC, and JC were investigated using XRD analysis. The analysis was carried out with the help of a Rigaku Miniflex-II diffractometer with a Cu-K-Alpha radiation ($\lambda = 0.15416$ nm) operating at a 30 mA current and a constant voltage of 40 kV. The morphological analysis of the JC was done by a high-resolution field-emission-scanning-electron-microscopy (FESEM) TESCAN-LYRA-3, Czech Republic. In addition, transmission electron microscope (TEM: JOEL, JEM 2100F) was used to analyses the morphological and diffraction features of the JC. The graphite structure of the carbon films was investigated by utilizing iHR320 HORIBA Roman spectrometer with a charge-coupled device detector and a 300-mW green (532 nm) laser at room temperature. An Al-K-alpha monochromatic x-ray source (ESCALAB-250Xi XPS-Microprobe from Thermo-Scientific, USA) was attached to an x-ray photoelectron spectroscopy (XPS) has been used to assess the elemental composition of the prepared electrode. The pores distribution and SSA were determined using a N₂ adsorption/desorption Brunauer-Emmett-Teller (BET) method and a Micrometrics analyzer (ASAP 2020).

3.6 Electrochemical measurements of JC

The electrochemical characteristics of the carbon materials under investigation were examined through the utilization of symmetrical two-electrode setups. The supercapacitor was immersed in a 0.1 sodium sulfate electrolyte for a duration exceeding 11 days. The

anode and cathode of the electrochemical analyzer were individually linked to the stainless-steel components, along with the connectors of the analyzer. The equations (1-4) were utilized to compute the specific capacitance of the electrode (C_{sp}), specific energy (E), specific power (P), and coulombic efficiency (η) values of the symmetrical supercapacitor based on the data obtained from the GCD analysis.

$$C_{sp} (Fg - 1) = \frac{2I\Delta t}{m\Delta V} \quad 3.1$$

$$E (Whkg - 1) = \frac{C_{sp}(\Delta V)^2}{28.8} \quad 3.2$$

$$P(Wkg - 1) = (E \times 3600)/\Delta t \quad 3.3$$

$$\eta = t_{\text{discharge}}/t_{\text{charging}} \quad 3.4$$

where m is the total mass of the active materials (g), I is the applied current (A), Δt is the discharge time (sec), and ΔV is the potential window (V).

3.7 X-ray diffraction (XRD)

To determine the major and minor compounds in our RM, XRD analysis was carried out on each RM-modified sample. The resulting pattern from the analysis is shown in Figure

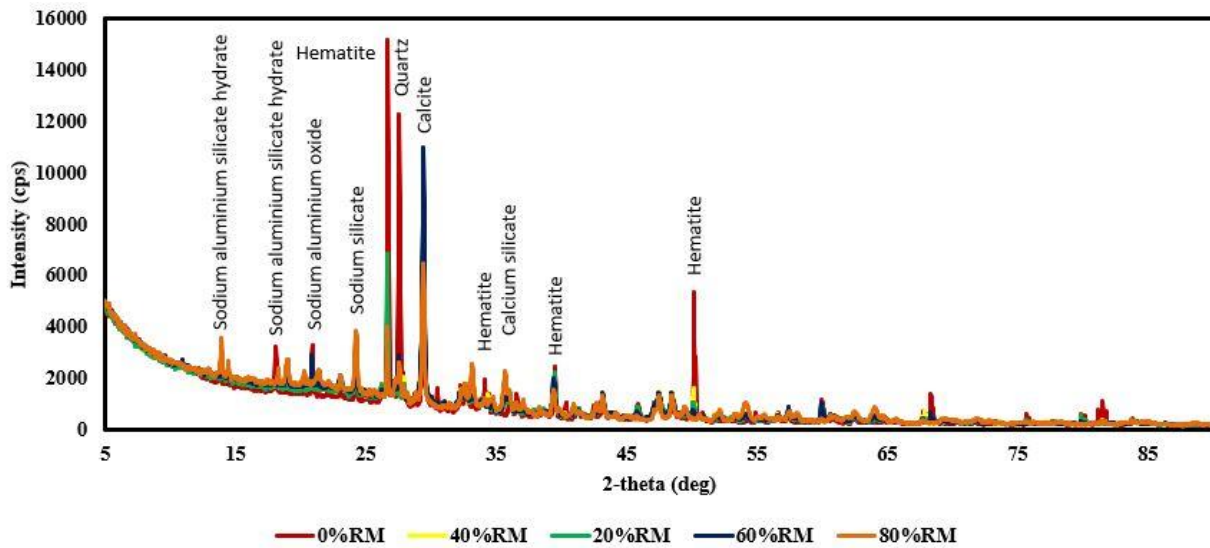


Figure 23 XRD results

2 and the detected compounds along with their formulas are shown in Table 2. Three major phases were detected in our RM, hematite, quartz, and calcite. Some of which were detected in various spots, such as the hematite at $\sim 27^\circ$, $\sim 33^\circ$, and $\sim 50^\circ$. Minor compounds, including sodium silicate, sodium aluminum oxide, and sodium aluminum silicate hydrate were also present. Pure RM seems to emit the highest hematite and quartz signal and, with the reduction of RM content, the signal seems to be diminishing. The results show that RM is rich in aluminum, iron, and calcium-based compounds, which comes in conformity with the literature. It is also noticeable that many of the detected compounds are in oxide form, as expected.

Table 7 The compounds present in RM and their formulas

Compound	Formula
Calcium silicate	CaSiO_3
Sodium aluminum silicate hydrate	$1.01\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 1.68\text{SiO}_2 \cdot 1.73\text{H}_2\text{O}$
Sodium aluminum oxide	NaAlO_2
Sodium silicate	Na_2SiO_3
Calcite	CaCO_3
Quartz	SiO_2
Hematite	Fe_2O_3

3.8 SEM

SEM analysis was also done to every set of mortar samples. The results are shown in Figure 24. The first picture, for the 0%RM samples, shows a lumpy microstructure, compared to the other samples. Clear gaps and pores are also shown in the body of the 0%RM sample. These pores seem to be diminishing with the addition of RM. This can be observed in the second picture (for the 20%RM) where the structure of the sample appears to be more intact and no visible gaps are observed from the same sampling level. The same trend continued with the addition of more RM and the pores seems to be completely vanished at 40%RM dosage. Further addition of RM reversed their effect, nonetheless, as the second row of figures depict. For instance, the 60%RM sample shows a grainy structure with small holes scattered around. The 80%RM sample, on the other hand, shows much larger lumps and a long gap, on the upper left corner, as well.

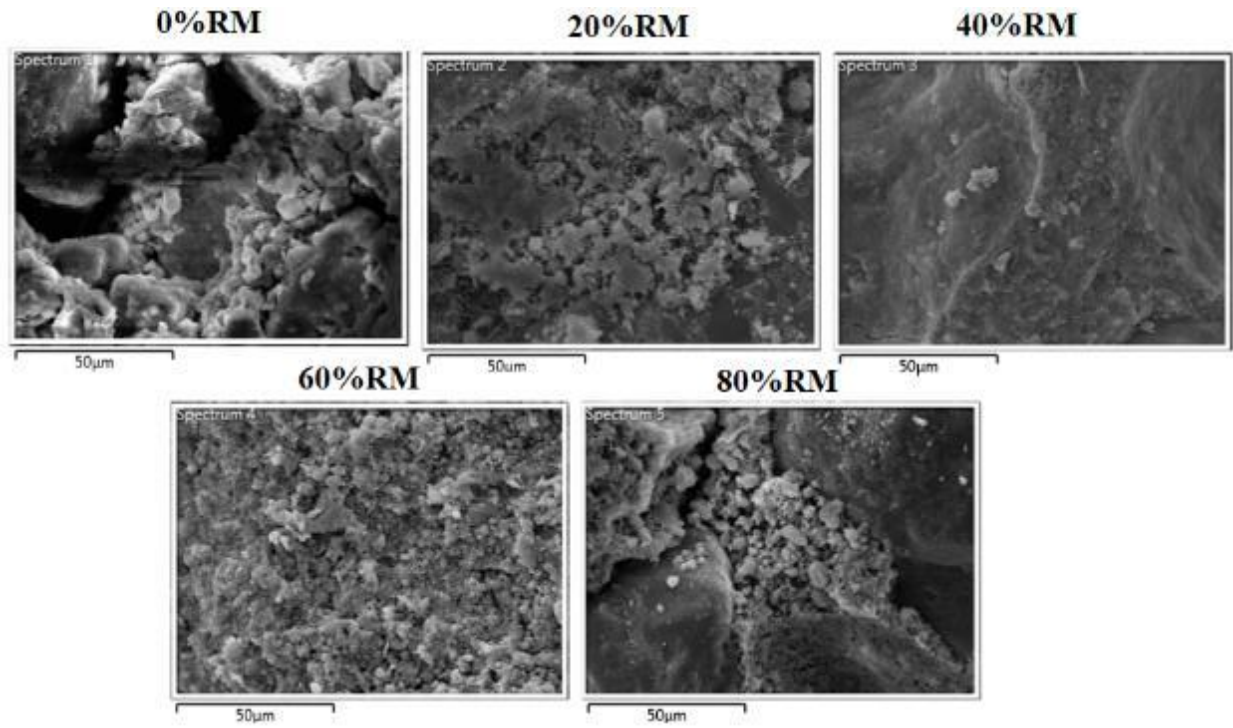


Figure 24 SEM results for RM-modified samples

3.9 XRF

Qualitative analysis of several elements was performed by XRF to measure its concentration in the RM used in this work. The analysis reveals the elements composing the analyte and their compounds as well. In case of the RM used in this experiment, XRF analysis showed a few major elements including aluminum, silicon, calcium, and iron. These elements detected here were also detected by A. Akincia. and R. Artir. There were also some minor elements detected such as phosphorus and potassium. The major and minor elements present in our RM along with their compound are presented in Table 8 and Table 9. The XRF analysis also revealed that the OPC is mostly composed of calcium (59%), as expected, and it also contains silicon at 23%. The remaining composites in the OPC matrix, as per the XRF analysis, are mainly aluminum and iron. The results showed that RM, however, contains a high concentration of aluminum (about 25%), silicon (16%), iron (15%).

RM also contains a small but noticeable amount of calcium and titanium (5% and 6% respectively). A comparison between the most significant elements in RM and OPC is presented in Figure 25.

Table 8 Compounds of the major and minor elements identified by the XRF analyses of RM

Compound	Element	Norm. Int.	Concentration	
Al ₂ O ₃	Aluminum	721.20	25.06	%
SiO ₂	Silicon	908.53	16.42	%
P ₂ O ₅	Phosphorus	39.37	0.29	%
S	Sulfur	287.48	6163.00	µg/g
Cl	Chlorine	200.18	2380.00	µg/g
K ₂ O	Potassium	164.85	0.06	%
CaO	Calcium	17776.38	4.69	%
TiO ₂	Titanium	35351.17	5.80	%
V ₂ O ₅	Vanadium	945.70	1091.00	µg/g
Cr ₂ O ₃	Chromium	1114.39	712.70	µg/g
MnO	Manganese	660.86	253.50	µg/g
Fe ₂ O ₃	Iron	61380.38	14.87	%
SrO	Strontium	11301.61	1784.00	µg/g
ZrO ₂	Zirconium	1713.79	1684.00	µg/g
Nb ₂ O ₅	Niobium	144.53	131.40	µg/g
Ce ₂ O ₃	Cerium	255.95	177.10	µg/g
Sum			68.71	%

Table 9 Compounds of the major and minor elements identified by the XRF analyses of OPC

Symbol	Element	Norm. Int.	Concentration	
Al ₂ O ₃	Aluminum	155.29	6.32	%
SiO ₂	Silicon	1197.15	23.46	%
P ₂ O ₅	Phosphorus	34.79	0.23	%
S	Sulfur	863.92	17150.00	µg/g
Cl	Chlorine	76.65	892.10	µg/g
K ₂ O	Potassium	1477.50	0.57	%
CaO	Calcium	180929.59	59.47	%
TiO ₂	Titanium	805.26	0.29	%
V ₂ O ₅	Vanadium	43.38	101.40	µg/g
Cr ₂ O ₃	Chromium	67.30	56.00	µg/g
MnO	Manganese	384.35	334.10	µg/g
Fe ₂ O ₃	Iron	6286.57	3.11	%
SrO	Strontium	2446.90	465.10	µg/g
Nb ₂ O ₅	Niobium	11.00	12.40	µg/g
Ce ₂ O ₃	Cerium	20.11	0.30	µg/g
Sum			95.48	%

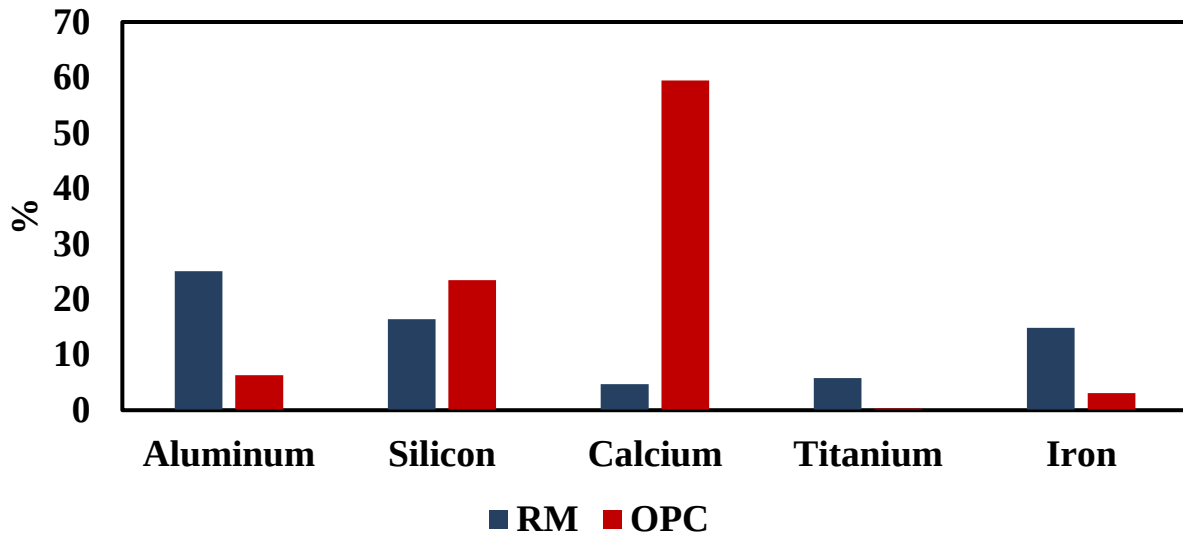


Figure 25 A comparison between the most significant elements revealed by the XRF in OPC and RM as a percentage

3.10 Compressive strength

The compressive strength test indicates that RM can affect the mortar's strength positively or negatively depending on its dosage. In the 20% RM replacement case, the strength was slightly increased from about 25 MPa to around 27 MPa. Further, an increase in RM content beyond 20% drastically decreased the compressive strength to reach a low of 6 MPa at 80% RM replacement. All the samples with RM content greater than 20% had a lower compressive strength than the control sample. This trend was common among all the 7-day, 14-day, and 28-day samples.

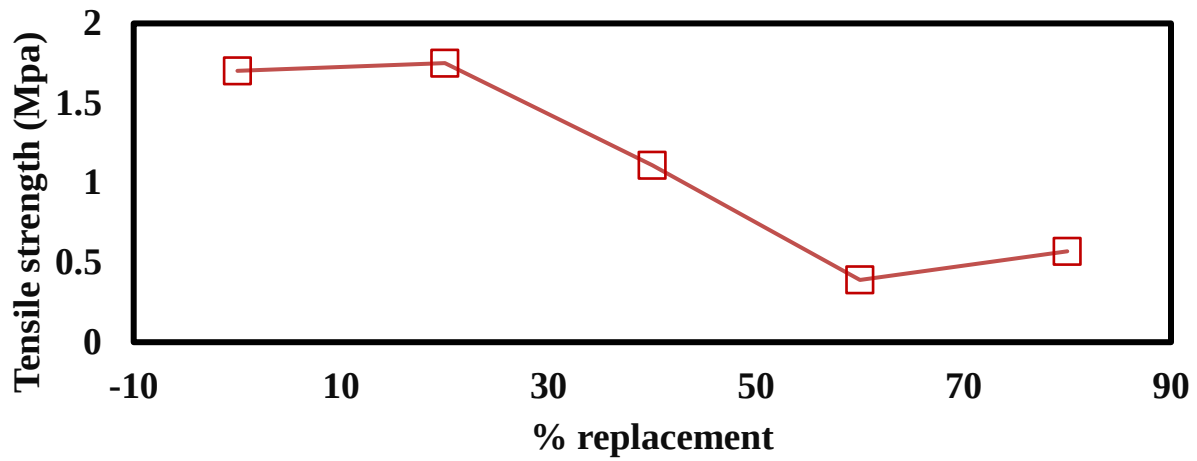


Figure 26 tensile strength results

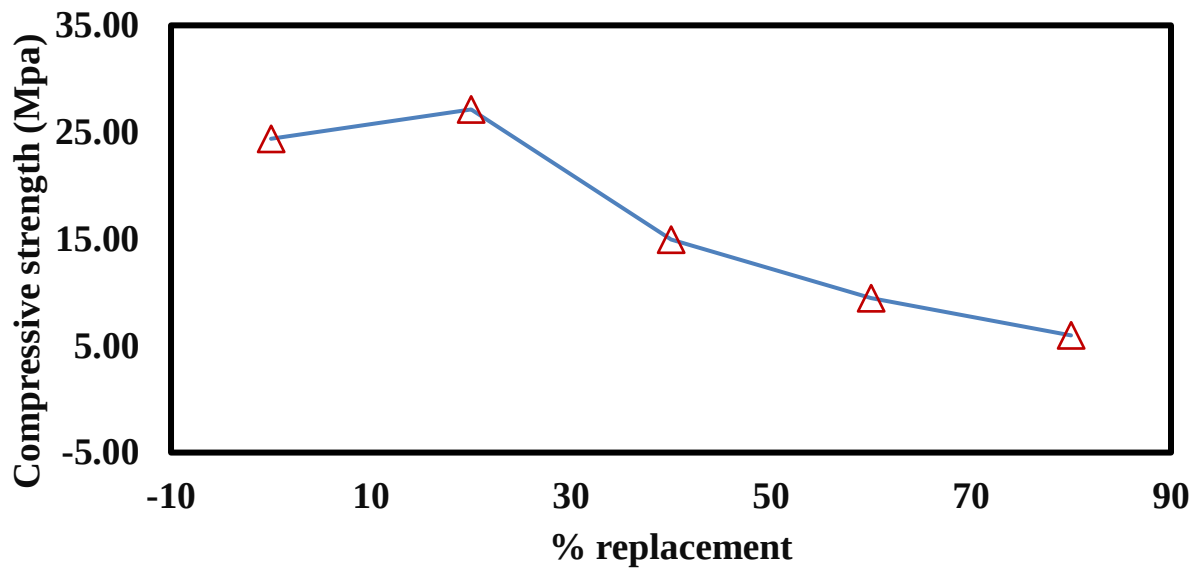


Figure 27 compressive strength results

This adverse effect of RM can be attributed to the lack of CaO in RM, which is the main component in cement, with a typical content of around 67% in normal cement. Conversely, RM usually contains a much lesser concentration of CaO, around 22%. However, a small

amount of RM increased the compressive strength slightly, which might be due to the filling-ability of RM. In addition, there are some elements present in both cement and RM besides CaO that help increase the cementing properties of RM. For example, SiO₂ can be found in both cement and RM at almost equal concentrations (around 20%). Al₂O₃ is also a common component between the two materials, but its concentration is much lower in cement (around 18% in RM and around 4% in cement). Nonetheless, the slight increase in the compressive strength with the addition of RM seems to be due to the changes in the microstructure of the mortar matrix when RM was introduced. This claim is supported by the results of the SEM test, where the figures show a more dense and compact structure with an increase of RM of up to 40%. With more RM, however, the mortar particles appear more agglomerated and form a lumpy structure. One possible cause is the lack of RM's cementing properties, which makes the samples with high RM dosage more fragile. This was noticeable even with the naked eye, especially with the 80% RM samples.

3.11 Tensile strength

The tensile strength of RM-modified mortars also increased slightly when a relatively small amount (20%) of RM was added. However, the increase was negligible, from 1.7 to 1.75 MPa. Similar to the compressive strength, the tensile strength dropped linearly to reach a minimum value of less than 0.4 MPa. The modulus of elasticity of the 20% RM samples was also significantly higher than all the other samples, which is not difficult to foresee by interpreting the compressive strength results.

3.12 Modulus of elasticity

The modulus of the control samples was around 19 GPa and then peaked at approximately 27 GPa when RM was added at 20%. At 40% RM, the modulus value was almost equal to 19 MPa, similar to the control sample. The figures for the elasticity modules continued to decrease with the addition of RM and reached 6 MPa at 60% RM. The value remained almost constant beyond the 60% point. Figure 26 and Figure 37 summarizes the compressive and tensile strength results, while Figure 28 shows the corresponding modulus of elasticity.

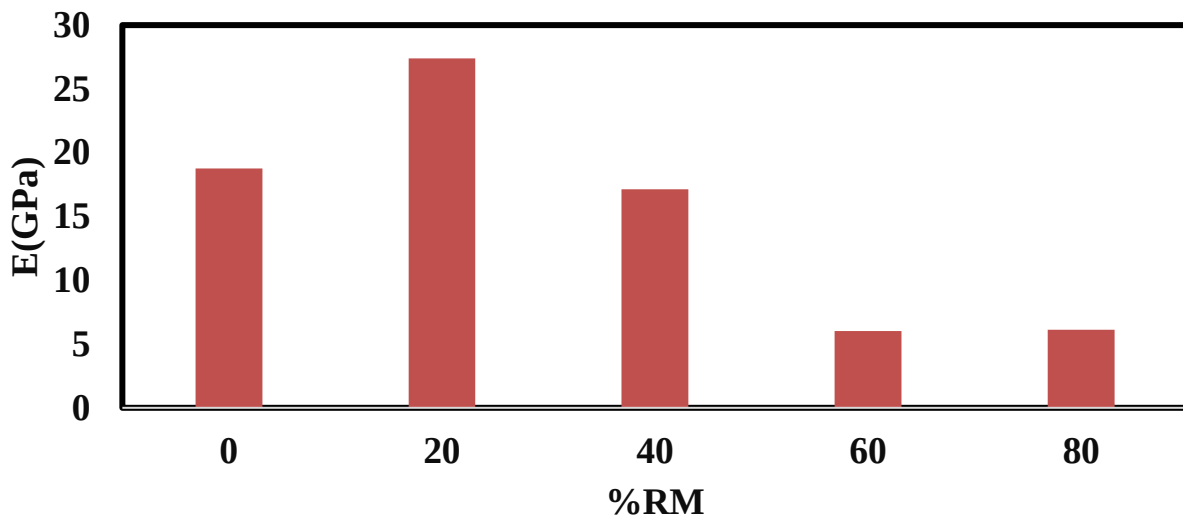


Figure 28 modulus of elasticity results

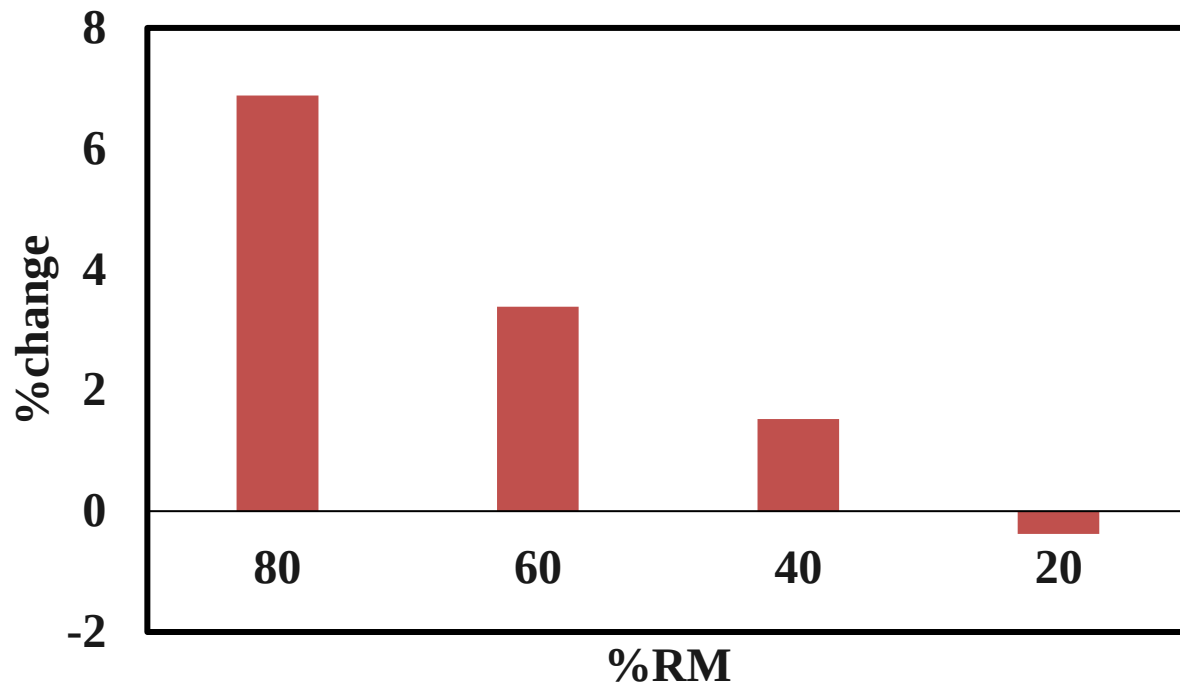


Figure 29 %change in RM sample weight due to water

3.13 Water absorption

The water absorption test shows that RM samples absorb more water than plain cement samples. Surprisingly, however, adding RM by 20% reduced the water absorption by less

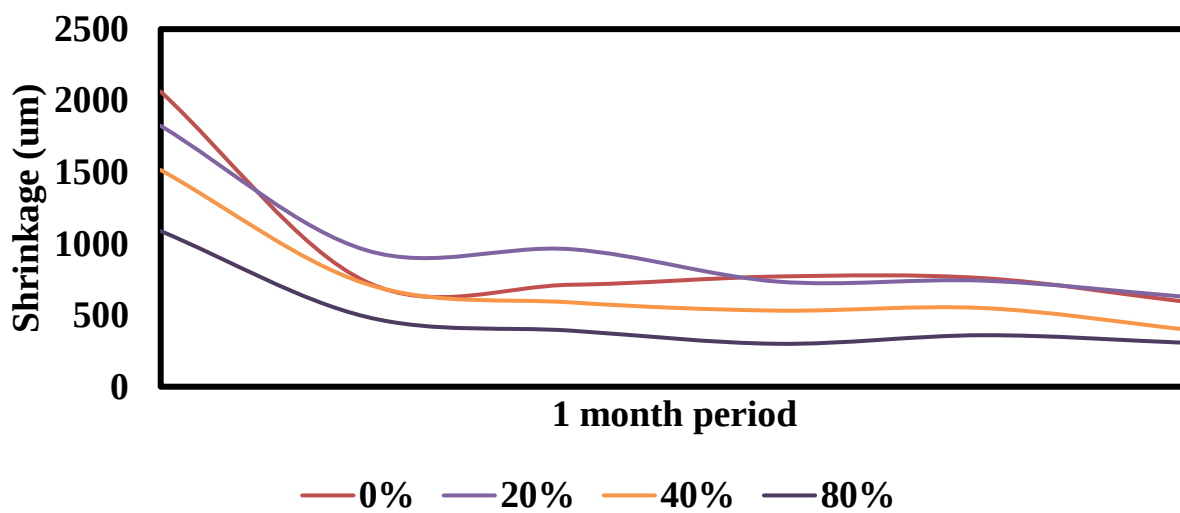


Figure 30 shrinkage results

than 0.5%, which might be considered within the margin of error. Increasing the RM content to 40% increased the sample weight by around 2%, and the weight gain continued to increase. The 60% RM specimens gained around 4% extra weight from absorbing water, and the 80% samples gained around 7% extra weight compared to the control sample submerged in water for the same duration. The result is shown in Figure 29.

3.14 Shrinkage

The shrinkage was also measured for all the samples, and the results followed a common trend. That is, the rate of change in the length of the sample was rapid in the first few days and then gradually slowed down to become almost linear. The period at which the rate decreased was the same for all the samples. The addition of 20% RM only slightly affected the shrinkage, and the final value of shrinkage, measured around a month from the start of the tests, was the same for the control and 20% RM samples. However, the test indicates that adding more RM can reduce the overall shrinkage because the 80% RM specimens had a lower final shrinkage value compared to the other samples. In addition, the rate of change of shrinkage became more subtle with the addition of extra RM. The result is shown in Figure 30.

3.15 Electrochemical characterizations

After hardening, the sample was placed in a sodium sulfate solution for several days before starting the electrochemical tests to ensure full penetration of the solution. The sample was kept moist during the test by wrapping it in filter paper soaked in sodium sulfate. The electrodes were also separated with weight paper to prevent electrical discharge. Figure 31 shows the 3*3*3.9cm RM supercapacitor. The supercapacitor was subjected to four different electrochemical tests: cyclic voltammetry (CV), galvanostatic charge/discharge (GCD),

electrochemical impedance spectroscopy (EIS), and cyclic stability. Two different voltage windows were considered: one ranging from -0.5 V to 1.5 V, and the other ranging from 0 V to 1.5 V. The scan rates for the tests ranged from 0.1 V/s to 0.4 V/s.



Figure 31 The test sample used in the electrochemical testing

Two voltage windows were conducted for the CV and GCD analyses: 0 to 1.5 V, and -0.5 to 1.5 V. Multiple cyclic voltammetry (CV) analysis was conducted at scan speeds ranging from 10 to 40 mVs⁻¹, while GCD analysis was performed at current densities ranging from 0.015 to 0.076 Ag⁻¹. The as-prepared sample (AC) underwent a cyclic stability test by subjecting it to repeated GCD cycles for a maximum of 400 cycles. The current density used was 5 Ag⁻¹, and the potential range was between -0.5 and 1.5 V. The electroencephalogram (EIS) was conducted at room temperature using an open circuit potential (OCP) within the frequency range of 100 kHz to 1 MHz, with an alternating current (AC) amplitude of 10 mV.

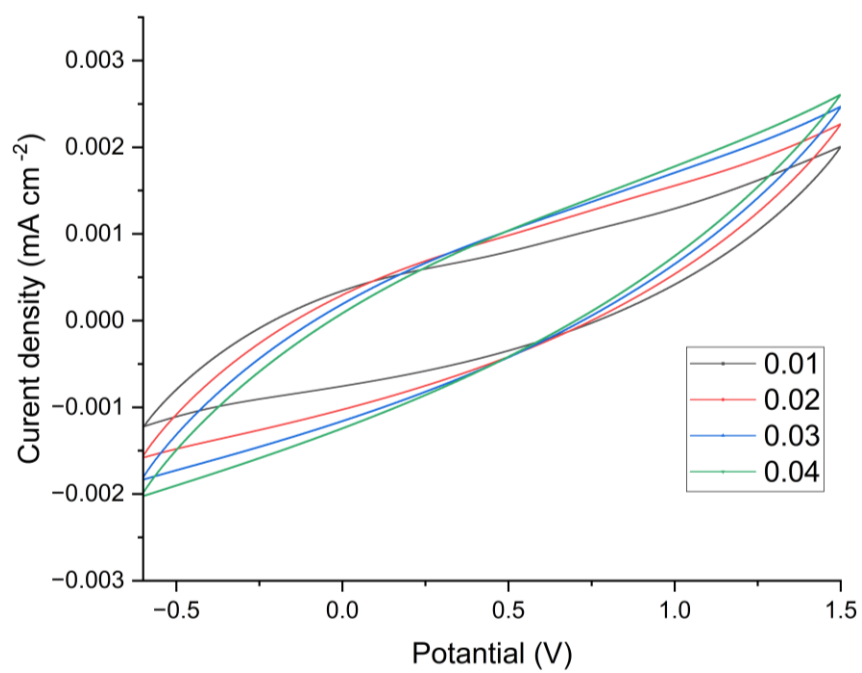
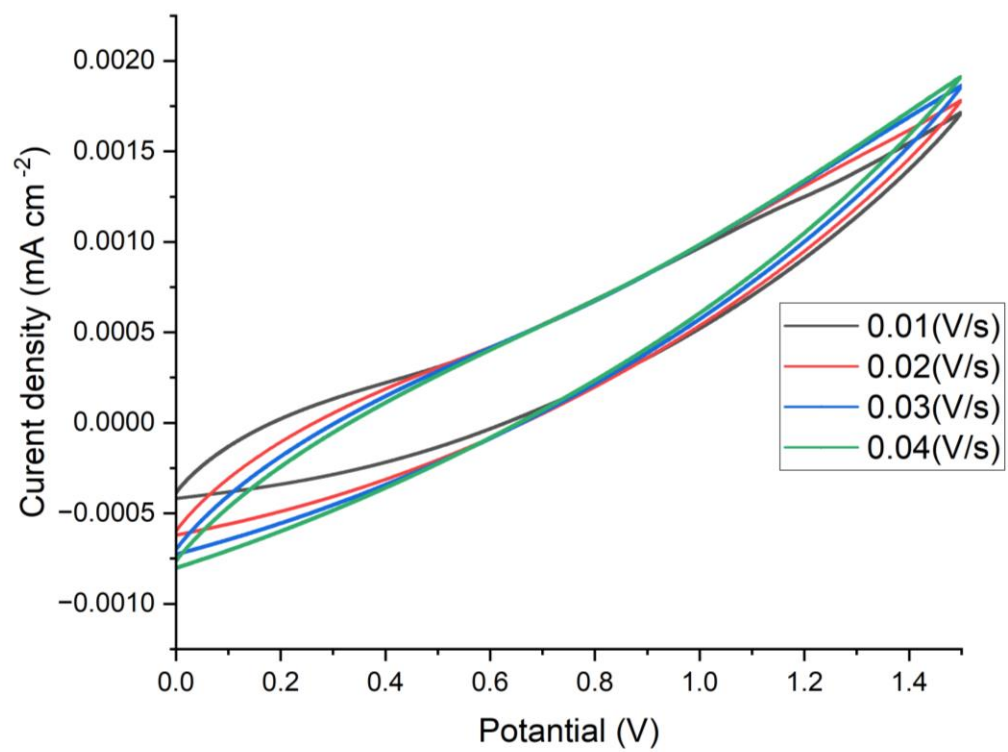


Figure 32 CV test with a different scan rate for two different volt window

The galvanostatic charge/discharge (GCD) test, shown in Figure 33, was carried out at different currents ranging from 0.0004 A to 0.001 A at an increment of 0.0001 A and 0.002 A as well. Two voltage windows were considered for this test as well. The first of which spanned from 0 V to 1.5 V and the second one spanned from -0.5 V to 1.5V. The specific capacitance value for the former was 2.34 F/g and 5.859 F/g for the latter. This comes in conformity with the CV test results. The highest capacitance was obtained at the lowest current (0.0004 A) for both voltage windows and the lowest capacitance was obtained at the highest current (0.002 A). The plot with the wider voltage window (-0.5 V to 1.5 V) yielded more accurate results compared to the one with a narrow voltage window due to its superior discharge curve and better intermediate resistance drop.

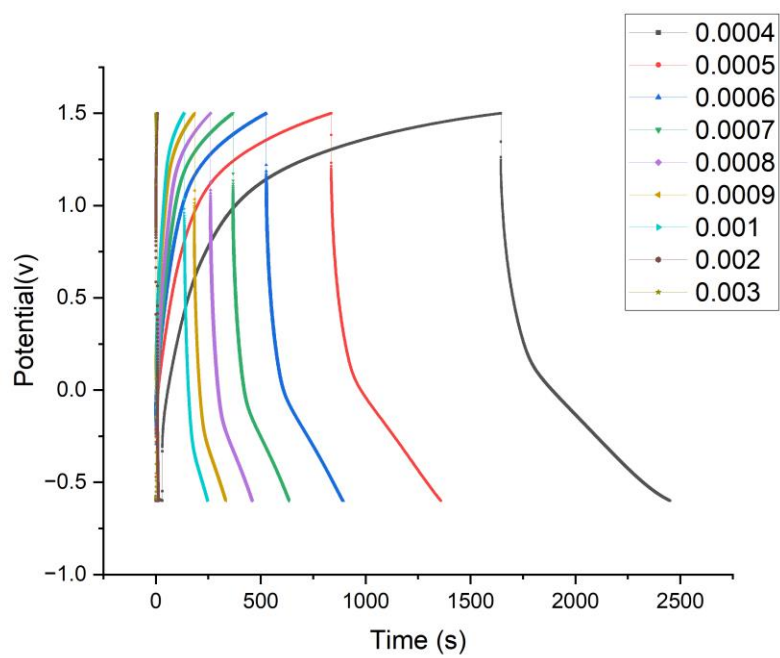
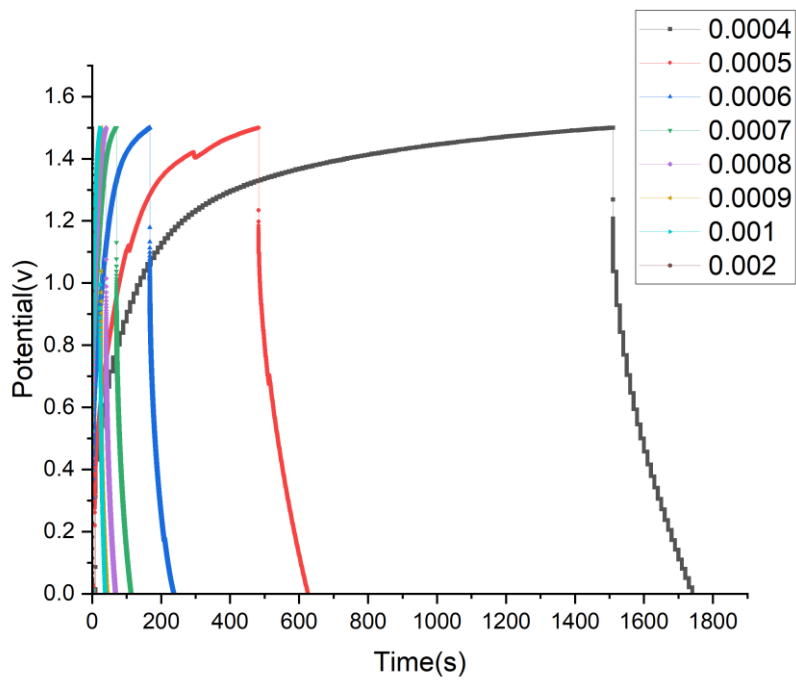


Figure 33 GCD test with a different current for two different volt window

To assess the performance of the selected electrode material, a cyclic stability test was performed. The supercapacitor was subjected to 1000 charge/discharge cycles and the retained capacity was recorded at 100 cycles intervals. The voltage window was set from -0.6 V to 1.5 V. The results, illustrated in Figure 34, show that the RM supercapacitor lost 10% of its capacitance in the first cycle and another 10% during the second cycle. The capacitance drop was steady and linear during these first two cycles. After that, however, there was no performance degradation and the capacitance retention remained almost constant (at 80%). There was only a minor drop ($< 3\%$) in the capacitance after the third charge/discharge cycle. These results show a good performance and reconfirm that RM is a potential supercapacitor material and reflect a good capacitive nature of the developed RM-based supercapacitor in addition to the robustness and durability of the supercapacitor under repeated charge-discharge cycles. It is also noteworthy that the separator and electrolyte materials also contribute to the cyclic performance of supercapacitors.

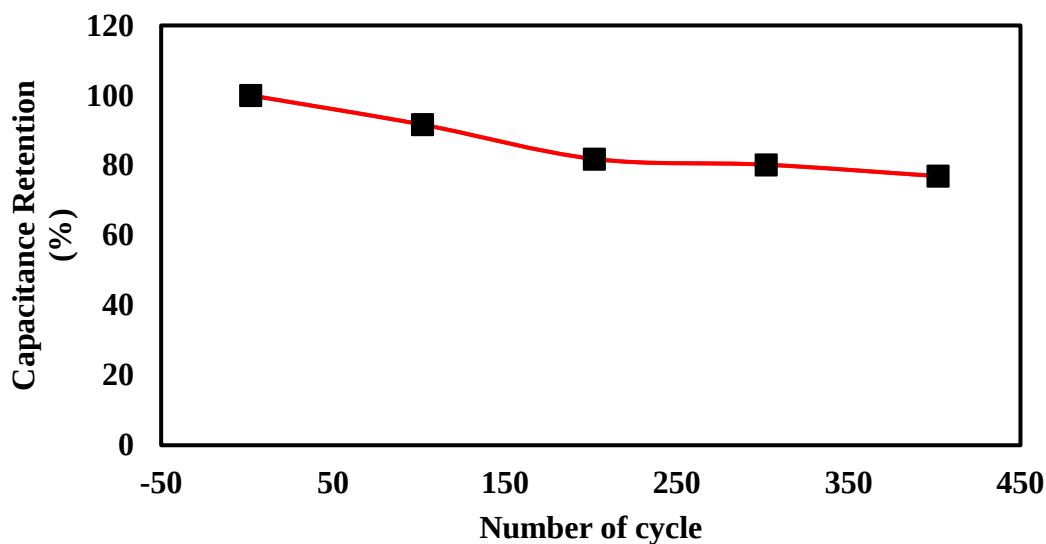


Figure 34 Stability test for the 400 cycles of supercapacitor

Another parameter that is used to measure the performance of supercapacitors is the power density, which characterizes the rate at which a device can release the energy stored, while energy density represents the amount of energy that it can contain. These values are calculated using the following equations:

$$E = 0.5 \times C \times \frac{V^2}{3.6} \quad (\text{Wh kg}^{-1}) \quad 3.5$$

$$P = \frac{E \times 3600}{t} \quad (\text{W kg}^{-1}) \quad 3.6$$

Here, E denotes the specific energy (Wh kg⁻¹), C is the specific capacitance (Fg⁻¹), V signifies the potential window (V), P is power density (W kg⁻¹), and t indicates the discharge time (s). Figure 35 portrays the correlation between energy density and power density for two distinct voltage windows. The first graph with a 0 - 1.5 V voltage window shows a steep abrupt decrease in the energy density followed by a gradual and steady reduction. The energy density measured 0.75 Wh/kg at 10 W/kg and dropped to 0 Wh/kg at almost 60 W/kg. The drop in energy density was almost 0.6 Wh/kg in the 10 - 30 W/kg power density range, but only dropped by 0.1 Wh/kg in the 30 - 60 W/kg range. The second graph with a wider voltage window (-0.6 V to 1.5 V) shows a very similar trend where the energy density rapidly decreased from 3.5 Wh/kg to almost 0 Wh/kg in the 0 - 40 W/kg power density range then remained constant in the 40 - 120 W/kg range.

Figure 36 demonstrates that an increase in current density led to a concomitant decrease in capacitance. This trend was shared among both the voltage windows. The capacitance – current graph for the 0 – 1.5 V window shows a slight discrepancy where the capacity

increased when the current increased from 0.02 to 0.04. This, nonetheless can be considered an error in the results and it was only observed in this instance. The other graph (with a -0.5 - 1.5 V voltage window) doesn't show this error and the specific capacity was observed to decrease linearly with the increase in the current density. In the case of -0.5 - 1.5 V, the rate at which the capacitance changes seemed to be constant until a specific threshold current value was reached; about 0.03 A/g in this particular experiment. The capacitance decreased steeply until the value was reached then dropped suddenly to almost zero. The 0 - 1.5 V shows a less intense reduction in the specific capacity with the increase in the current. The figures also show that a max capacitance of about 2.5 F/g was obtained at 0.01 A/g in the 0 - 1.5 V range while the highest capacitance of 5.859 F/g was attained under a current density of 0.015 A/g with a voltage window ranging between -0.6 and 1.5 volts while the specific capacity in the 0 - 1.5 V voltage range peaked at approximately 2.5 F/g for the same current. The capacitance dropped to zero at about 0.03 A/g in the -0.5 - 1.5 V range and at 0.08 A/g in the 0 - 1.5 V range.

The LED test is commonly conducted as the final experimental test on a supercapacitor. The LED test in the supercapacitor lasted for around 5 minutes, which is an excellent result, and it demonstrates the supercapacitor's ability to deliver energy to an external load.

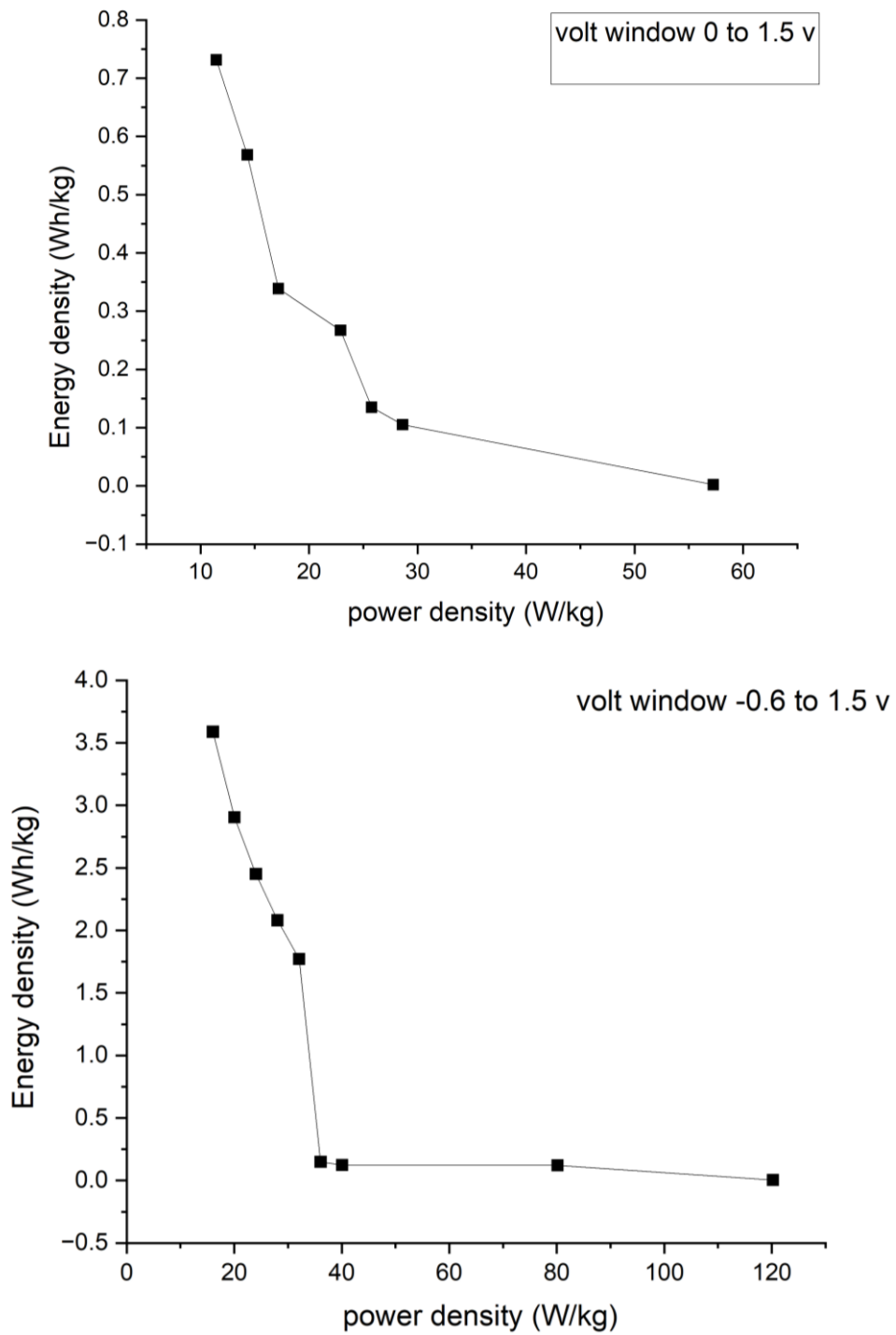


Figure 35 power density vs energy density for the supercapacitor

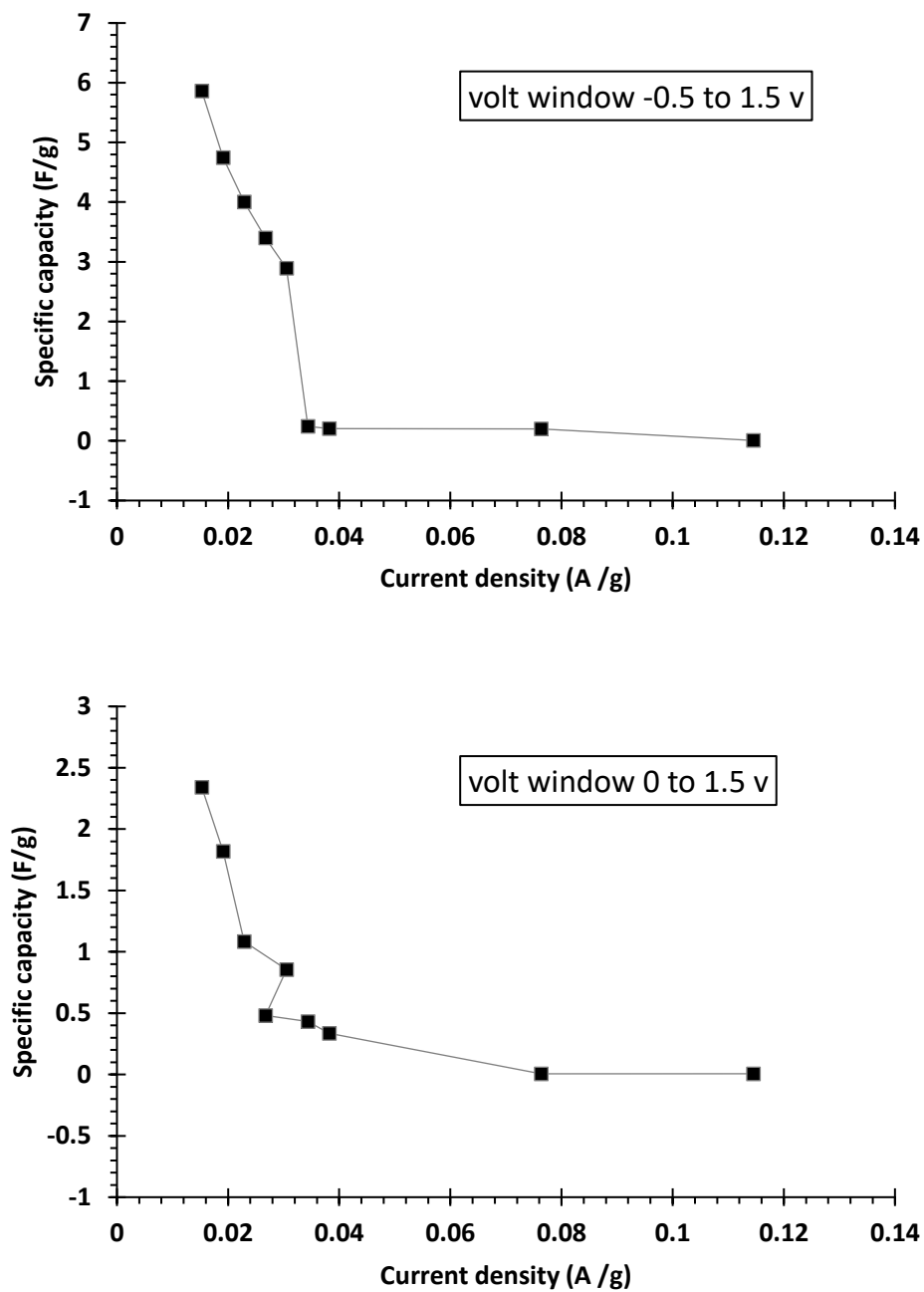


Figure 36 Capacitance vs current density for two different voltage windows

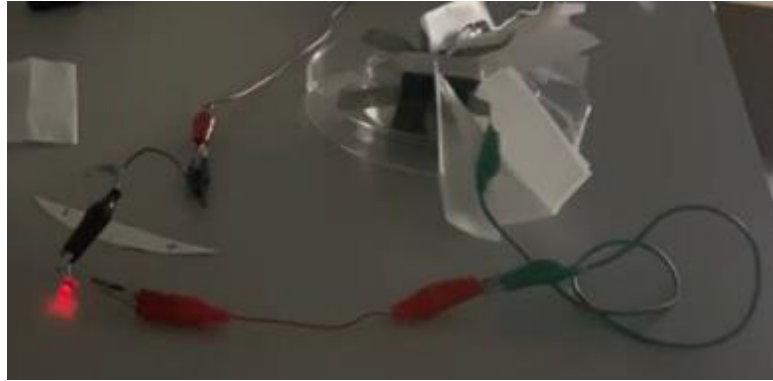


Figure 37 LED test

3.16 Conclusion

this study assessed the potential use of the alumina production by-product (red mud) as a part of cement-based structural supercapacitors. The data and results from this experiment add to the field of knowledge of this developing industry. This study presented many outcomes. Firstly, an exhaustive assessment of the composition and micro-structure was provided and it was found that RM has the potential to be used as a structural supercapacitor. Secondly, the performance of RM-modified mortars was assessed against straining actions and durability hazards. This stage of testing showed that 20%RM replacement can sometimes perform better than plain OBC mortars in certain areas. Finally, the electrochemical tests proved that the developed supercapacitor, which is fabricated from RM, cement, sand, water, jute derived carbon, and steel meshes, has an acceptable electrochemical performance. Overall, the results indicate that RM-modified mortars can certainly be used as structural supercapacitors.

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