STRUCTURE AND RAMAN SPECTROSCOPIC STUDIES OF SOME CONSTRUCTION MATERIALS

M.Sc. Thesis

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Structure and Raman Spectroscopic Studies of some Construction Materials

A THESIS

Submitted in partial fulfillment of the requirements for the award of the degree of Master of Science

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CANDIDATE'S DECLARATION

I hereby certify that the work which is being presented in the thesis entitled **STRUCTURE AND RAMAN SPECTROSCOPIC STUDIES OF SOME CONSTRUCTION MATERIALS** in the partial fulfillment of the requirements for the award of the degree of **MASTER OF SCIENCE** and submitted in the **DISCIPLINE OF PHYSICS, Indian Institute of Technology Indore**, is an authentic record of my own work carried out during the time period from August 2020 to May 2022 under the supervision of Prof. Rajesh Kumar and Prof. Sandeep Chaudhary

The matter presented in this thesis has not been submitted by me for the award of any other degree of this or any other institute.

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ABSTRACT

Waste accumulation has subsequently increased to dangerous levels. Some of these wastes are tyre rubber waste and industrial wastes i.e., Brick powder (BP), Marble powder(MP), Concrete powder(CP), and Fly ash(FA). Their disposal is a serious matter of concern so their recycling would be advantageous. It has been studied that use of rubber in mortar deteriorates the hardened properties of the mortar. In this experimental work waste tire rubber is used as a fine aggregate with 10% replacement of sand along the industrial wastes as recycled powder as a replacement of cement from 0% to 30% to produce an environmental friendly mortar. The characterization of raw materials is carried out by XRD and Raman Spectroscopy . Even the industrial wastes are tested for pozzolanic activity by calculating Strength Activity Index. The mechanical properties of the Recycled Powder Self Compacting Mortar with rubber aggregate(RPSCM-R) are tested by compressive strength test, flexural strength test and calculating the water absorption and porosity. The microstructure analysis of the RPSCM is carried out by Raman Spectroscopy and XRD . It was found that the degraded hardened properties due to the use of rubber as a fine aggregate can be compensated by the use of recycled powders as a replacement of cement.

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Chapter 1

Introduction

Cement production has undergone tremendous developments since its beginning some 2000 years ago[1]. The production of cement is an energy-intensive process that requires thermal energy. In the process, solid particles heat up to 1450 °C and cool The process results in CO_2 -rich exhaust streams[2]. Cement is made from limestone. It is heated to form limestone, which is Calcium Oxide emitting Carbon di-oxide. Approximately 750 kg (1650 lb) of CO_2 is emitted for each tonne (2205 lb) that is made. The production of cement is the cause of around 7% of the total greenhouse gas emissions.

There are different no. of ways which have been proposed to solve the problem like carbon capture and inventing completely new concrete but the only significant method to reduce greenhouse gas emissions is the use of "cement replacements"[3]. Demand for the cement worldwide was about 2.283 billion tons in 2005, 2035 million tons in 2007, and 2836 million tons in 2010 with an increase of 130 million tons annually. The total cement production worldwide for 2016 was about 4.2 billion tons. Even the energy consumption for cement production for cement and solve the for 3.6 to 6.5 GJ/ton depending on process and location of production[4].

Production of cement worldwide has increased around 20% in the past decade. Production of cement approximately conssumes around 110 KWh/tonne. Globally, production of cement uses 18.7TWh which is 2% of primary energy consumption of the world and 5% of energy consumption by industries[5].

Recycled powders such fly ash, brick powder, marble powder, concrete powder are classified as waste and is even considered harmful to the environment. Even the tire rubber waste which is increasing almost linearly with the increasing production of vehicles and it has been reported that world's annual tire production exceeded 2.9 billion tires anually in 2017. And it's disposal is again a matter of serious concern[6].

Burning tire or using it as a fuel cause the emission of toxic gases. Moreover, as tire contains styrene, a strongly toxic substance which is highly damaging for humans so it's dumping can be highly damaging for humans[7].

Fly Ash which is formed due to the combustion of pulverized coal(IS 3812: Part 1,2013), is considered a fine waste. If the fly ash is disposed in an open environment it would result in the degradation of soil as well as ground water. Particles of fly ash can even spread through the medium of air can cause a serious risk to health. Similarly, if marble waste is left unrecycled it will cause environmental pollution as well as ground water contamination[6]. Moreover, the significant way to recycle Construction and Demolition Waste is using the clay brick waste powder as supplementary cementitious material in concrete/mortar.

Considering the environmental concern , one of the best ways to reduce CO_2 emission is to reduce the use of cement . Different types of waste can be used in concrete which can help preserve the environment and reduce the cost of construction , by using them as a replacement of cement .

For larger projects which need a lot of cement pozzolanic materials play a key role in concrete as a partial replacement of cement .These pozzolanic materials could be Construction and demolition waste powders such as Brick Powder, Marble Powder, Concrete Powder, Fly Ash etc. Clay Bricks are very widely used for building throughout the world .Earthquakes, reconstruction and demolition of buildings results in the accumulation of waste bricks [8] . Usual method to tackle construction and demolition waste is through landfill , which is quite expensive and requires a larger area . So, broken waste bricks can be used as an aggregate and as a partial replacement of cement which we are going to explore in the following research work.

As a construction material marble has been prominently used for a very long time .Owing to it's great use ,management of marble waste has become an issue of great concern. It causes a great environmental damage because of it's increased stockpiles on the roadside and abandoned lands [9].

Material Additions which can behave as filler or pozzolana or cementing agents can decrease the quantity of clinker in cement Usually these materials are residues obtained from other processes like cow dung waste fom cattle rrearing, silicon waste from used solar panels. Considering the environmental impact these wastes decreases the clinker percentage per m^3 of cement and is good in at least two ways i.e., Cutting off emissions of CO₂ and recycling waste products[10].

Chapter 2

LITERATURE REVIEW

In the experimental study, Anitha Selvasofia S.D. et. al.[11] examined the effects on the concrete mechanical properties with marble powder as fine aggregate . It was demonstrated that concete mix demonstrated excellent workability, compressive strength and split tensile strength upto 10% replacement of WMP in concrete. Recently, an innovative method called " paste replacement method" was given by reserchers. Using this method , waste marble is added to replace cement paste(water + cement) keeping water to cement ratio constant Li et. al. ,2018[12] proved that durability and dimensional stability of mortar improved significantly and also, content of cement can be reduced upto 33% .

Aliabdo et. al. ,2014[13] performed experiments in which cement and sand is replaced `by 0%, 5%,7.5%, 10%, and 15% marble powder by weight for w/p = 0.5 and w/c = 0.40. The analysis performed by various methods like TGA, XRD and SEM had shown improvements in the mechanical characteristics of concrete. Uysal et. al. ,2012[14] conducted study on fresh and hardened properties of SCC by using fly ash, limestone powder, basalt powder and marble powder to replace cement. Even Aliabdo et. al. ,2014[13] reported 15% increase in splitting tensile strength when 10% marble powder replaced cement and sand amd reported that the resulting concrete mix exhibited higher performance relative to thayt of conventional concrete.

Vishal Behl et. al. 2021[15], conducted study of the properties such as water demand, heat of hydration for fresh concrete, durability of hardened concrete, permeability etc. and compared with fly ash based concrete mix. A good compressive strength was reported in 10% fly ash concrete mix. N. Rohith et. al. ,2022[16] performed tests on concrete mixes prepared by using copper slag(CS) as partial replacement of cement to compare the fresh and hardened properties with conventional concrete. Flexure, Split tensile strength and Compressive Strength test were Dcarried out to study the hardened properties with the conventional concrete. Optimum percentage turnned out to be 30% fly ash and 30% copper slag as a replacement of fine aggregate and cement respectively. Ayad Al-Yusuf et. al.,2021[17] have reported that the effect of fly ash(FA) content as replacement of cement on compressive strength and fracture toughness of the

concrete and reported that 17% fly ash content (by weight) as optimum percentage for fracture toughness and it was also reported that improved compressive strength and fracture toughness can be obtained at 20% cement replacement with fly ash.

Veera Horsakulthai et. al., 2021[18], investigated porosity, compressive strength, electrical resistivity and water absorption of self-compacting mortar with recycled concrete powder used as a replacement of cement. The compressive strength, porosity, and coefficient of water absorption for 20%, 40%, and 60% recycled concrete powder at 28 days are given as follows :

Cement replacement		Compressive Strength	Porosity increment	Water	Absorption
With	Recycled			Coefficient	Ī
Concrete Po	wder				
20%		77.1MPa	6.3%	34.4%	
40%		56.6MPa	16.5%	70%	
60%		37.7MPa	26.6%	149.3%	

All the mentioned properties were studied for 20%, 40% and 60% recycled concrete powder as areplacement of cement . And the compressive strength has shown decreasing trend whereas porosity and water absorption had shown increasing trend .

Dae-Joong Moon et. al. ,2005[19] however reported that the flexural and compressive strength of the mortar with the waste concrete powder at 7 and 28 days were quite lesser than that of the conventional concrete because of the dehydrated cement within the waste concrete powder. Yong Jic Kim et. al. , 2012[20] have tried to utilize waste concrete powder as a recycled aggregate .He reported that as the percentage replacements of cement by WCP increased there is a decrement in the flow value by 30% and the compressive strength has shown reduction of about 73% and the increment of about 70% in the sorptivity coefficients.

Trilok Gupta et. al., 2014[21] reported that the use rubber in the concrete reduces the mechanical properties such as flexural and density of the concrete . Literature has revealed that rubber fibres reduces the mechanical properties of the concrete wheras the use of recycled powder enhances them. This research work is an exploration for using waste tire rubber and the recycled powders simultaneously in mortar to produce a greener(environmental -friendly) mortar with minimum loss or developed mechanical properties.

Chapter 3

EXPERIMENTAL PROGRAM

3.1. Materials

Two types of mortars with four different recycled powders are prepared using rubber aggregate and brick powder, marble powder, concrete powder, and fly ash.First type of mortars are prepared by using the four recycled powders as replacement of cement with four recycled powders i.e., brick powder, marble powder, concrete powder, and fly ash which are Recycled Powder Self Compacting Mortar (RPSCM) specimens . Another type of mortars are prepared by using the four recycled powders along with rubber aggregate as replacement of sand by 10% which are Recycled Powder Self Compacting Mortar with Rubber Aggregate(RPSCM-R) specimens.

3.2. Mix proportion

Both types of specimens are prepared using the 10%,20% and 30% recycled powders as a replacement of cement. The designations used are shown in Table 1:

Recycled Powder	% Replacement	Designation for	Designation for
		RPSCM	RPSCM-R
No Recycled Powder	0%	0RP	RPOR
Brick Powder	10%	BP10	BP10R
	20%	BP20	BP20R
	30%	BP30	BP30R
Marble Powder	10%	MP10	MP10R
	20%	MP20	MP20R
	30%	MP30	MP30R
Concrete powder	10%	CP10	CP10R
	20%	CP20	CP20R
	30%	CP30	CP30R
Fly Ash	10%	FA10	FA10R

Table 1: Designations of the mix proportions

20%	FA20	FA20R
30%	FA30	FA30R

In this work, the SCM was prepared at 1:1.5 volumetric proportion of cement to the fine aggregate. The constant water to cement ratio of 0.35 was adopted. For RPSCM mixes, the dose of superplasticiser was adjusted to obtained the desired slump flow (range 240-260 mm) according to the EFNARC [22]. The mixes prepared in this work are shown in Table 2. In the Table 2, the former letters such as BP, CP, FA, and MP denote brick powder, concrete powder, fly ash, and marble powder, respectively. The following number represents cement replacement at 10%, 20%, and 30% levels. A SCM mixes with no cement replacement was considered as reference mix and designated as RP0.

The SCM mixes were prepared in a rotary pan-type mixer. A dry-to-wet mixing approach were adopted for uniform blending of the mix.

Mix ID	Cement	Recycled powder	Fine aggregate	Rubber fiber	w/b ratio	Superplasticiser
			k	g/m^3		
Series I	1					
0RP	738.06	-	1310.33	-	0.35	0.36
BP10	664.26	59.23	1310.33	-	0.35	0.40
BP20	590.45	118.46	1310.33	-	0.35	0.50
BP30	516.64	177.69	1310.33	-	0.35	0.60
CP10	664.26	53.55	1310.33	-	0.35	0.32
CP20	590.45	107.11	1310.33	-	0.35	0.45
CP30	516.64	160.66	1310.33	-	0.35	0.60
FA10	664.26	60.18	1310.33	-	0.35	0.20

Table 2: Quantities of materials for recycled powder self-compacting mortar (kg/m3)

FA20	590.45	120.35	1310.33	-	0.35	0.23
FA30	516.64	180.53	1310.33	-	0.35	0.25
MP10	664.26	76.68	1310.33	-	0.35	0.30
MP20	590.45	153.36	1310.33	-	0.35	0.32
MP30	516.64	230.04	1310.33	-	0.35	0.35
Series II						
RPOR	738.06	-	1179.29	53.27	0.35	0.36
BP10R	664.26	59.23	1179.29	53.27	0.35	0.40
BP20R	590.45	118.46	1179.29	53.27	0.35	0.50
BP30R	516.64	177.69	1179.29	53.27	0.35	0.60
CP10R	664.26	53.55	1179.29	53.27	0.35	0.32
CP20R	590.45	107.11	1179.29	53.27	0.35	0.45
CP30R	516.64	160.66	1179.29	53.27	0.35	0.60
FA10R	664.26	60.18	1179.29	53.27	0.35	0.20
FA20R	590.45	120.35	1179.29	53.27	0.35	0.23
FA30R	516.64	180.53	1179.29	53.27	0.35	0.25
MP10R	664.26	76.68	1179.29	53.27	0.35	0.30
MP20R	590.45	153.36	1179.29	53.27	0.35	0.32
MP30R	516.64	230.04	1179.29	53.27	0.35	0.35

3.3. Testing methods

3.3.1. Tests on raw materials

Strength Activity Index(Determining Pozzolanic Activity)

The strength activity index of the four recycled powders (i.e., Brick Powder, Marble Powder, Concrete Powder, Fly Ash) were calculated in accordance with ASTM C311[23] to determine the pozzolanic activity. The control sample was prepared by mixing 500g OPC cement, 1375g graded standard sand, and 242mL of water. Test samplaes were prepared by replacing 20% OPC cement with the four recycled powders. The water to cement ratio was altered to obtain similar flow values as that of control sample . Mortar samples were cast into 50mm cubes and were demolded after 24h and placed in a curing tank for 7 and 28 days for compressive strength . The Strength Activity Index is calculated by the following equation 1

$$SAI = (A/B)*100$$
 (1)

Where A= average compressive strength of the test mix cube ,B=average compressive strength of the control mix

XRD characterization

The XRD characterization of the raw materials (recycled powders and OPC) was carried out on X-Ray Diffractometer with scattering angle variations from 5 to 90 degrees. The JCPDS files were used to identify different minerals in the samples of raw materials and the recycled powders

Raman analysis

The raman characterization of powdered samples of recycled powders and OPC(sieved from 90micron) was carried out in the range (100 -2100) cm^{-1} using a Raman spectrometer.

3.3.2. Hardened properties of RPSCM and RPSCM-R

Compressive Strength and Flexural Strength

The mechanical properties of RPSCM were studied by compressive strength and flexural strength tests. A compressive strength test was conducted on a cube specimen of $50 \times 50 \times 50$

mm at 28-days curing age following ASTM C109 [24] at a 0.150 MPa/s loading rate. A flexural strength test was performed on a prismatic beam of $40 \times 40 \times 160$ mm 28-days curing age following ASTM C348[25] at the constant load rate of 0.044 kN/sec.

Water Absorption and Porosity

Three mortar cubes for each of the RPSCM and RPSCM-R were casted having dimensions 70.6x70.6x70.6mm³ for calculating the water absorption and porosity.The tests were performed and calculations for the obtained data has been done in accordance with the code ASTM C 642[26]

Chapter 4

Results and Discussions

4.1. Characterization of raw materials

4.1.1. Strength Activity Index

The average compressive strengths of the control and test mixes at 7 and 28 days of curing are shown by the following table

Mix ID	At 7 days of curing	At 28 days of curing
Control mix	61.5 kN	75.1 kN
BP-20	62.7 kN	74.8 kN
FA-20	82.8 kN	85.2 kN
CP-20	65.5 kN	68.7 kN
MP-20	65.3 kN	83.0 kN

By using the equation 1, the strength activity index of Brick Powder, Fly ash, Concrete Powder and Marble Powder calculated after 7 days of curing are 101, 132, 106, 106 and after 28 days of curing are 99,99,91 and 110 respectively. According to ASTM C 311 the materials having SAI greater than or equal to 75 are pozzolanic active. Hence all four recycled powders are pozzolanic active.



4.1.2. XRD Analysis of Raw Materials





Brick Powder-XRD

Bricks are made by baking clay at(900-1100)°C .Clay is an alumina silicate and the formula is $Al_2O_3.2SiO_2.2H_2O$, But it is never found in pure form.There are several different clay minerals; for example Kaonilite – clay mineral with chemical composition $Al_2Si_2O_5(OH)_4$.

The following minerals are found at respective angles

MINERAL	2θ (in degrees)
Calcite	23,28,39,41
Quartz	21,27,41,42.5,51,55,60,68
Chlinochore	19
Muscovite	8.5,46
Sanidine	27.5

Marble Powder -XRD

Marble is a metamorphic rock composed of recrystallised carbonate materials most commonly

calcite or dolomite. The following minerals were found in XRD analysis of Marble Powder:-

MINERAL	2θ(in degrees)
Calcite	28,31,37.5,41,45,51
Copper	45
Natroxalate	31,37.5,41,45

Concrete Powder-XRD

Concrete is composed of cement ,fine aggregates and coarse aggregates mixed with water which hardens with time.It's XRD analysis reveals the following minerals :-

MINERAL	2θ (in degrees)
Anorthite	13,21
Albite	22,29
Calcite	27.5
Aluminium	39
Quartz	29,41,51

FLY-ASH (P_60) XRD

This fly ash is taken from Thermal power plant. It is a thermal industry based fly ash (also known as Coal Ash). The following mineral phase are identified in XRD analysis of Fly Ash(P_60):-

MINERAL	2θ (in degrees)
Quartz	21,51,27,38
Mullite	16,26,32.5,35,41,42.5
Haematite	35

OPC(Ordinay Portland Cement) -XRD

In the manufacturing of ordinary portland cement (OPC) at first ,Limestone is grinded with iron ore and heated at around 1500°C to form clinker which is then mixed with gypsum to form ordinary Portland Cement.The following phases are found in XRD analysis of OPC :-

MINERAL PHASE	2θ (in degrees)
Alite	32.5,29
Belite	32.5,29
Quartz	41
Calcium Carbonate(Calcite)	29,23

Calcium hydroxide phase appear probably due to slight hydration of OPC with atmospheric moisture



4.1.3. Raman Analysis of Raw materials







Raman analysis of Marble Powder(MP),Concrete Powder(CP),Fly Ash(FA) and Brick Powder(BP):-

The Raman Analysis of Recycled Powders and Fly Ash are found in agreement of the same upto a great extent . The minerals found and their corresponding shifts are

	BRICK POWDER	MARBLE	CONCRETE	
		POWDER	POWDER	
MINERALS	RAMAN SHIFTS (cm ⁻¹)			
Calcite	282,712	282,712,1086,437	1086	
Quartz	263,356,463,500			
C-S-H gel		_	263	
		_	1086,704	
gypsum	_	_	1156	

Fly Ash(P_60)_Raman

In this sample of fly $ash(P_{60})$, the mineral phases present could not be identified. It may be noted from EDX results that silica percentage is more in this coal-based fly ash. The presence of luminiscence only may be attributed to the presence of amorphous silica. [27]



OPC(Ordinary Portland Cement)_Raman

Phases of the cement in Raman Analysis found are Alite, Belite, C-S-H gel,Ettringite and Gypsum.Since gypsum is the major component in the manufacturing of cement hence it's prominent peak is quite expected.Since, the sample probably might have absorbed moisture from the atmosphere because of which it has undergone a little hydration and hence small peak of C-S-H gel has occurred.

The corresponding wave number of Raman shift of various phases are :

CSH gel	709 cm ⁻¹
C ₃ S,C ₂ S	$(812-868) \text{ cm}^{-1}$
Ettringite	991 cm ⁻¹
Gypsum	1085 cm ⁻¹

4.2. Hardened Properties

4.2.1. Compressive Strength



The compressive strengths of all the RPSCM-R are quite less than their corresponding RPSCM including the control mixes in which there is no replacement of cement i.e. 0RP and RP0R. This might be due to hydrphobic behaviour of rubber due to which voids are created near the rubber aggregates and hence the mortar mix formed is quite weakened. Similar phenomenon of debonding and cracks are reported in the research work of Laibao et. al.,2022 [28], at the interface between cement matrix and rubber aggregate which even accounts for weak interfacial bonding strength between them. This weakening in the rubberized concrete is reported by Su et.

al.,2014[29] Rubber being an organic material and hydrophobic in nature does not participate in the cement chemistry.

BP_mixes

The Brick powder blended mixes have shown an increasing trend in compressive strength upto 20% replacement of cement and therafter starts decreasing .Similar trend goes for both RPSCM and RPSCM-R. The trend is quite unmatched upto 20% with the reults reported for the rubberized concrete by Yueqing Gao et. al. , 2022[30] but with further replacement the observations are in line.As the EDX results of the Brick powder indicates the amount of silicates and alumina are quite higher for brick powder which decreases Ca/Si ratio and improves Al/Si ratio which in turn favours the formation of CASH more than C-S-H in the brick powder blended mortar as a replacement of cement . It has been reported by Lothenbach et.al. ,2019 [31] that CASH causes more denser packing as compared to CSH . Formation of CASH occurs by the following reaction

 $CH + A + S \longrightarrow C-A-S-H$

(Portlandite) (Alumina) (Silica) (Calcium Aluminate Silicate Hydrate)

The above reaction seems to be increasingly favoured upto 20% replcement . Afterwards, with the increase in cement substitution with Brick powder, the availability of formed portlandite decreases , which limits the formation of C-A-S-H. Hence, the increment in strength is observed upto 20% and thereafter it starts decreasing.

MP_mixes

The compressive strength of the mortar specimen cubes comprising 0%, 10%, 20% and 30% marble powder as a replacement of cement is observed to increase upto 10% and thereafter decrease . It might probably be due to decrease in the Alite(C₃S) and Belite(C₂S) phases which accounts for mortar strength . Similar results were reported by Aliabdo et. al. , 2014[13]. One of the possible reasons for improvement in compressive strength could be the filler properties of marble powder. Even Ergun, 2011[32] reported improved compressive strength with waste marble powder as 5% replacement of cement . Further, 15% replacement of cement with marble waste showed reduction in compressive strength.

CP_mixes

The corresponding compressive strengths of the mortars with 10% ,20% and 30% concrete powder as replacement of cement have shown almost similar or it seemed a slight increment in compressive strength. The improvement in the strength with reference to control mix i.e., RP-0 is probably due to hydration of the unhydrated cement in the concrete powder leading to the formation of additional CSH which provides a denser and durable mix. Even Veera Horsakulthai et. al., 2021[18] studied impact of recycled concrete powder strength and reported that the compressive strength decreases with increase in cement replacement with the concrete powder. The optimum percentage of concrete powder was found to be 20%. But , the compressive strength of the RPSCM-R showed a decreasing trend which is probably due to the uncompensated voids.

FA_mixes

With the cement replacement by fly ash in the amount of 10%, 20% and 30% the compressive strength obtained are 52.91MPa, 47.43MPa, and 47.4MPa respectively. The positive effect on the compressive strength is due to the formation of C-S-H(Calcium Silicate Hydrate) and C-A-H(Calcium Aluminate Hydrate) which provides density and durability . Again, the decrease in compressive strength with 20% and 30% replacement of cement with fly ash is probably due to decrease in the availability of adequate cement which in turn reduces the lime generation and hence reduces the amount of strength gained due to pozzolanic reaction. Therefore, overall reduction in the compressive strength is observed .Similar trend of decreasing compressive strength is observed by My Ngoc-Tra Lam et. al., 2018[33] and Gudmundur Hanneson et. al., 2012 [34] However, trend reversal is observed in the RPSCM-R of FA_mixes and with the increasing replacement of cement , the compressive strength increases.

4.2.2. Flexural Strength

Flexural strength is the ability of the material to withstand bending forces applied perpendicular to it's longitudnal axis. As we can see from the graphs shown below even the flexural strengths of the RPSCM-R have values lower than that of RPSCM which can be attributed to the same reason that the voids and cracks created by the presence of the rubber aggregates results in the poor interlocking between the rubber aggregates and the cement matrix. The hydrophobic nature

of rubber might also cause the less formation of primary hydration products and secondary hydration products which is another reason for the poor compaction of the matrix. This decrease in flexural strength is reported by Trilok Gupta et. al.,2014 [21].



BP_mixes

The flexural strength of the mortar beams formed due to replacement of cement by the brick powder in the amount of 10%, 20% nd 30% replacement of cement had shown similar trend as that of compressive strength with highest flexural strength at 20% replacement of cement due to brick powder. It has been reported in the literature that cement substitution with the brick powder improves dynamic elastic modulus and hence flexural strength Yueqing Gao et. al., 2022[30].

Replacement beyond an optimum percentage reduces the dynamic elastic modulus and also limits the pozzolanic reactions which explains the trend. As rubber aggregate is not participating in the cement chemistry the trend for both RPSCM-R and RPSCM is same and the optimum percentage to replace cement by Brick powder even for flexural strength is 20%.

MP_mixes

Marble powder has abundance of Calcium rich phases and provides better sites for nucleation of

the hydrated phases. Even Calcium rich content facilitates the formation of secondary hydration products such as C-S-H along with lime formed due to primary hydration of cement. Therefore, a significant positSive effect on flexural strength was observed upto 10% replacement of cement with marble powder. Thereafter, a decreasing trend in the flexural strength is observed in MP-20 and MP-30 mixes. The trend observed are in line with those reported by M.A.Rashwan, 2020 et. al., 2020[35] but do not match with R.Rajkumar et. al., 2020 . However, with reference to control specimen i.e., RP-0 of the RPSCM-R the flexural strength MP-10 rubber mix decreases. This is probably because the voids and cracks are widened due to the tendency of the marble powder to form all hydration products around itself which increases with the amount of marble powder as replacement of cement .

CP_mixes

The considerable increase in the flexural strength of the concrete powder blended mixes is due to the additional C-S-H which is formed due to concrete powder in the CP mixes. This improvement of flexural strength is observed upto 10% replacement of cement with concrete powder .This is probably due to less availability of lime to form secondary hydration products .Even the primary hydration products decrease due to less cement. The trend is somewhat similar for both RPSCM and RPSCM-R.Yueqing Gao et. al. , 2022[30] reported the similar results for RPSCM with concrete powder for flexural strength.

FA_mixes

Flexural strength of the fly ash blended specimens is found to increase upto 20% replacement of cement with fly ash and further replacement decreases the flexural strength which matches with the results reported by Sanchit Gupta et.al.,2022 [36]. This behaviour is probably because fly ash

contains thermally activated mineral phases which favours the formation of C-S-H which in turn (provides more flexural strength as compared to Portladite($Ca(OH)_2$). The formation of C-S-H occurs more as lime available in cement undergoes pozzolanic reaction with fly ash upto 20% replacement of cement . Further, the replacement causes reduction in lime content and the amount of C-S-H formation reduces.



4.2.3. Water Absorption and porosity



Water absorption is the ability of water to migrate into pores. It is measured by measuring the increase in mass as a percentage of dry mass. Whereas, Porosity is the measure of the number of voids and microcracks in concrete. Through water absorption only the volume of the considerably bigger voids can be calculated where the water can easily penetrate whereas through the porosity calculation in which we put mortar specimens in boiling water the smaller voids and cracks can be included. The water absorption and porosity of the various RPSCM and RPSCM-R can be shown through the following graphs. Again , the porosity and water absorption of the rubberized mortar specimen i.e. RPSCM-R are both less than that of it's corresponding RPSCM.

BP_mixes

Brick is indeed fired clay so the Brick powder is rich inn silica content and it has considerable pozzolanic activity as calculated by Strength Activity Index test which indicates that pozzolanic reactions occur between the Brick powder and $Ca(OH)_2$. It promotes the formation of sperical C-A-S-H. It is reported by Xu Lao et. al.,2022 [37] and Arif Rehan et.al. 2021 [38]. So , the porosity and Water absorption remains almost similar upto 10% replacement of cement in RPSCM . Thereafter, the porosity starts increasing as the effective water available after primary hydration cement is more which after evaporating from the cement matrix leaves more voids and

hence increased water absorption and more Porosity. Moreover, the reduced amount of cement reduces lime generation and hence limits the occurance of pozzolanic reactions. But, there is only a slight increase in the BP_mixes with rubber aggregate as brick powder has a tendency to cause dispersion and dissolution of cement providing numerous nucleation sites for C-S-H gel causing voids and cracks around rubber to get widened.

MP_mixes

As marble powder is rich in Calcium so provides better nucleation sites for hydration which disperses the hydrated phases in the cement matrix improving the cement matrix and with higher replacement hydration of cement increases and size of the voids decreases which decreases the water absorption and porosity. Mohammad Rafi et. al., 2022 [39] and Yamanel Kenan et. al., 2019 [40] reported similar results. However, the reason for the decrease in the initial value for water absorption and porosity with 10% replacement of marble powder cannot be given as there is less well-defined literature on the reaction of carbonates. Almost the same trend is being followed by both RPSCM and RPSCM-R for water absorption. But, the porosity values of the rubber mortar specimens increases with further replacement of the cement with marble powder .

CP_mixes

Concrete powder might have several dormant phases which might get activated due to crushing, grinding and sieving . And, the unhydrated cement might also undergo hydration resulting in additional hydration products. But, that hydration will not probably be able to compensate the hydration caused by the primary hydration of the cement . Therefore with the increasing replacement of cement by the concrete powder the overall hydration products in the cement matrix decreases as a consequence of which the water absorption and porosity of the CP_mixes decreases for both RPSCM and RPSCM-R

FA_mixes

Fly ash has thermally activated mineral phases and has a high pozzolanic activity and with increasing cement replacement the secondary hydration products increases which decreases the voids and the packing becomes denser and denser. As a consequence of which the porosity and the water absorption of the RPSCM decreases .So, we got a decreasing trend for fly ash blended

mortar mixes for porosity and water absorption for both RPSCM and RPSCM-R. Even the hydrated phases are increasingly formed which is even reflected by the chigh compressive and flexural strengths of FA_mixes. Similar results are reported by Swathi Vennam et. al.,2022 [41].

4.3. Microstructure analysis of RPSCM

4.3.1. XRD Analysis

XRD Analysis Of Fresh Mortar Mixes



ORP -XRD

0RP is an ordinary Portland cement based control mixture (without any recycled powder) and is used as a reference mortar specimen for the study of blended mixtures i.e. Recycled Powder self-compacting Mortar(RPSCM). The result of the XRD analysis of the reference mortar specimen 0RP is shown in the figure. The following minerals were detected in 0RP:

MINERAL PHASE	2θ (Degrees)
Alite	29,32.5
Belite	31 , 32.5
Quartz	26.65 , 50.14 ,21 , 61
Portlandite	18,34.10,47,50.81
C-S-H gel	29.09
Calcite	29.3 , 29.4



BP_30 (fresh)-XRD

Brick is actually fired clay and the peaks of Quartz are quite expected to occur. Phases of Alite and Belite occur as the reactions in the cement have not yet occurred to a considerable extent.Since, Brick powder is a good pozzolanic material so the the small peaks of Ettringite and Portlandite occurs as the early hydration products after 24 hours of curing. Calcite peaks appear as it is the main ingredint of cement



MP_30(fresh)-XRD

Marble Powder is majorly composed of limestone($CaCO_3$) so the dominant peaks of Calcite are quite expected. Alite and Belite phases appear as the occurrence of reactions in the cement might not have undergone to a significant extent. However, Portlandite appears as the early hydration phase aftrer 24 hours of curing in the marble powder blended mix with 30% replacement of cement . Quartz phases occur due to the presence of sand.



CP_30(fresh)-XRD

Concrete powder is basically construction and demolition waste which might have some percentage of unhydrated cement . Moreover Calcite (CaCO₃) present in the cement takes part in the chemical reactions of cement . Even some of the unhydrated cement undergoes hydration reactions . Portlandite , Ettringite and C-S-H gel appears as the early hydration products . Alite and Belite phases appear as the main course of reactions have not occurred to a great extent. Calcite in the form of lime is the main ingredient of cement.



FA_30(fresh)- XRD

Fly ash has thermally activated phases and has high pozzolanic activity as compared to others. It participates in the chemical reactions of cement actively and during the early stage of curing it is observed that reactions do occur quickly and Portlandite and C-S-H gel are the phases which appears as the phases of hydration. C-S-H provides better hardening to the mortar specimen as compared to Portlandite.









BP-30 (28 DAYS)

Several peaks of hydrated phases like calcium Silicate Hydrate (CSH), Calcium Hydroxide(CH) and Calcium Sulfoaluminate Hydrate (CSAH) which again imparted great mechanical performance to 28 days cured Brick powder blended mixture BP-30 (28 days). Peaks of Quartz are found because of river sand

MP_30 (28 DAYS)

Marble powder is a Calcium rich industrial waste which promotes hydration and provides better nucleation sites in the cement matrix . So, we can get several hydration products in the mable powder blended mix formed as a replacement of cement such as C-S-H gel, Calcium Aluminate Hydrate , Calcium Sulfoaluminate hydrate , and Portlandite . The secondary hydration products predominantly provides considerable mechanical properties to the mortar . Calcite probably appears due to unreacted marble powder or due to the carbonation of Portlandite

CP-30 (28 DAYS)

Number of peaks of portlandite are found which were more in count and prominent in intensity than that of 0RP ,which is possibly due to the formation of $Ca(OH)_2$ as only hydration product. This provides densification to the concrete mix CP-30 and responsible for higher mechanical performance than 0RP.Presence of Quartz can again be attributed to river sand.Calcite phase also have several peaks which is probably due to carbonation.

FA -30 (28 DAYS)

Presence of Quartz phase was primarily due to sand particles. Intensity peaks of $Ca(OH)_2$ are found lower as compared to OPC based control mixture (ORP), which might be due to dilution of cement paste.

Replacement of cement with fly ash generally reduce the clinker phases(i.e. C_2S and C_3S),resulting in lower generation of $Ca(OH)_2$. Maximum intensity peak of CSH ,exhibits higher intensity peak of CSH , exhibits higher intensity peak of CSH gel than ORP. The CSH gel generally dominates the mechanical performance of concrete matrix.

This actually imparts higher mechanical performance to fly ash blended mixture (FA-30). More peaks of Ettringite are found as compared to 0RP that is generally responsible for densification of concrete matrix. The peak might not be that much prominent because of :Non-crystalline nature or Conversion of Ettringite into Calcium Monosulphate phase

4.3.2. Raman Analysis



Raman Analysis of fresh mortar mixes







BP-30(fresh)_Raman

Brick powder is prepared by burning clay which is mainly composed of SiO_2 (Quartz). So, we got three phases in Raman Analysis of brick powder blended mix i.e. Quartz,Ettringite and Calcite.Calcite appears as it is major component of cement. And,Ettringite appears as the only hydrated phase.

MP-30(fresh)_Raman

Marble powder is majorly composed of lime(CaO) with minor amounts of Silica, Magnesium Carbonate, Iron oxide, Aluminium Oxide, etc. The peaks of Calcite are quite expected. Besides, that phases of Quartz and Ettringite are also found. Ettringite's presence occurs as a major hydration product which is responsible for stiffening and hardening. As Marble powder is a high lime-containing material which facilitates the production of calcium aluminates which on further reaction with sulfates form Ettringite

CP-30(fresh)_Raman

Recyled concrete Powder (RCP) is obtained by mechanical treatment (crushing,grinding and sieving) from construction and demolition wastes(CDW). CDW has higher reusability ,lower cost and legal requirements and it is easy to obtain with high fineness and strong reactivity. The phases detected in Concrete powder blended mix are Calcium Hydroxide and Quartz. The peak of Calcium Hydroxide is quite weak as it might take time for the chemical reactions to progress and hydration phases to occur.

FA-30(fresh)_Raman

Fly ash is a great pozzolanic material with thermally activated mineral phases and quickly starts reactions with the cement mineral phases and produces secondary hydration phases which imparts significant mechanical properties to the mortar mix. However, the portlandite is the only hydrated phase which is detected in the raman analysis of Fly ash blended mix.



Raman analysis of cured mortar specimens







0RP_Raman

ORP is the reference mortar specimen with respect to which we are analysing the mechanical performance of the other mortar. The phases which are found are Calcite, Calcium Hydroxide and Quartz. Calcite is an important ingredient of ordinary Portland Cement. Calcium Hydroxide is found as the only hydrated phase.

MP-30(28 days)_Raman

Even after 28 days of curing no significant changes are observed in the phases of Marble powder blended mixture. Only peak of Ettringite becomes prominent which provides further hardening.

CP-30(28 days)_Raman

The concrete powder blended mix after 28 days of curing is detected with quite small and broad peaks of Belite(C_2S), Calcite, Ettringite and Quartz. This might be possible due to poorly crystalline structures of the phases.Formation of Ettringite can be considered to play a major role in providing the mechanical performance to the mix in comparison to the reference mortar specimen ORP. Calcite peaks are found as it forms major component of OPC.

BP-30(28 DAYS)_Raman

After 28 days of curing other hydration phases also appeared i.e.,CSH gel and Portlandite along with Ettringite. These hydration product provide considerable compressive strength and flexural strength to Brick powder blended mixture. Besides that,phases of Calcite and Quartz were detected.

Chapter 5

Conclusions

- All the four recycled powders i.e., Brick powder, Marble powder, Concrete Powder, and Fly Ash are pozzolanic active which is due to the significant presence of the phases like silica, alumina, and calcite.
- 2) The optimum percentage for the use of Brick powder, Marble powder, Concrete powder, Fly ash as a replacement of cement along with rubber aggregate for obtaining better compressive strength are 20%, 10%, 10% and 30% respectively.
- 3) The optimum percentage for the use of Brick powder, Marble powder, Concrete powder, Fly ash as a replacement of cement along with rubber aggregate for obtaining better flexural strength are 20%, 20%, 10% and 20% respectively.
- 4) The optimum percentage for the use of Brick powder, Marble powder, Concrete powder, Fly ash as a replacement of cement along with rubber aggregate for minimizing porosity are 20%, 10%, 10% and 30% respectively.
- 5) The microstructure study of the RPSCM reveals that presence of hydrated phases (C-S-H, CASH, CAH and portlandite) are responsible for better compaction and improvement in the mechanical properties of the mortar.

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