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Microstructural And Elemental Characterization Of Cement Mortar Modified With Marble Waste As Paste Replacement Using Taguchi Technique

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***Abstract**: This study investigates at the microstructural and elemental properties of cement mortar blends that have been changed by adding different amounts of marble waste as a paste replacement. The Taguhi method is used to plan experiments, find the best signal-to-noise ratio, and find the best means. Analysis of Variance (ANOVA) is used to study the effect of process parameters. We used scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDAX) to to examine morphological changes and chemical composition within the modified mortar matrices look at how the modified mortar matrices changed shape and what chemicals they contained. The best results come from replacing 10% of the marble waste, using 0.8 W/C, and letting it cure for 90 days. SEM analysis showed that the microstructure became denser over time and that more hydration products formed with the presence of marble waste. EDAX confirmed that important oxides like CaO, SiO_2, and Al_2O_3 were present. The findings show that marble waste is a good material to use instead of cement because it helps with hydration, micro-filling, and pozzolanic activity.*
***Keywords:** Marble Waste, Cement Mortar, Taguchi Technique, SEM, EDAX, Microstructure, Sustainable Construction.*

1. INTRODUCTION

Mortar, which is usually made of cement and sand, is the main material used to build masonry. The widespread use of cement and sand, while useful, is bad for the environment. Making cement uses a lot of energy and adds a lot of CO_2 to the atmosphere. On the other hand, too much sand mining harms river ecosystems and uses up natural resources[1]. Because of these problems, people are actively looking for more environmentally friendly alternatives to traditional mortar components. One way to do this is to use marble waste, which is what is left over after cutting and polishing marble stones, as a partial replacement for the cement paste in mortar. Using marble waste as a filler can lower the amount of cement needed, which is better for the environment, saves resources, and makes good use of industrial waste. This study looks into replacing marble waste for the cement paste without altering the water to cement ratio and to make cement mortar more environmentally friendly without affecting its structural performance. With the increase of marble slurry substitution in cement concrete and mortar mix, a reduction in consistency, air content, and compressive strength is noted while a increase in initial setting time, and soundness[2]. Studies have demonstrated that using marble waste in concrete can make it stronger and last longer. Replacing up to 15% of the cement in reinforced concrete with marble slurry has been shown to make it work better [3], [4]. Marble powder (MP) as a mineral substitute in mortar in a 1:3 ratio, with 10% replacement did not differ appreciably in strength or workability from the reference mix. But when MP was used instead of sand, the compressive strength dropped by 10%, and when it was used instead of cement at a later age, it dropped by 20% [5]. MP affects air-cured blended cement, the porosity raised with the MP, reaching 15% at 3, 7, 28, and 65 days. The compressive strength, on the other hand, went down by up to 14% at 28 days. Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray spectroscopy (EDX) showed that replacing cement with MP increased the calcium hydroxide (CH) content and decreased the C-S-H content. This made the air-cured blended cement paste more porous.[6], [7], [8]. SEM, EDX analysis, porosimetry, and nanoindentation revealed that using waste marble sludge instead of Portland cement up to 15% by weight made pastes stronger in both compression and bending and less porous [9]. It was observed that replacing 10% of the cement with marble powder didn't impact the properties of the mortar in terms of setting, soundness and strength compared to the reference mix.[10]. Up to 20% replacement of cement with marble dust showed improved resistance to corrosion and water absorption and compressive strength was not affected[11], [12]. From above studies we can see that using marble waste as a filler can lower the amount of cement needed, which is better for the environment, saves resources, and makes good use of industrial waste. This study looks into replacing marble waste for the cement paste without altering the water to cement ratio and to make cement mortar more environmentally friendly without affecting its structural performance.

2. MATERIALS AND METHODS

2.1 Materials

Ordinary Portland Cement (OPC) 53grade with fineness of 3.4 and specific gravity of 3.18 conforming to IS: 12269-1987 was used. Locally available M-sand passing through 4.75mm sieve, with fineness modulus of 3.3 and specific gravity of 2.63 was used as fine aggregate. Marble waste powder was obtained from industrial processing units and sieved to pass through 90 μm with specific gravity of 2.38, fineness modulus of 4.8 and rodded bulk density of $1000 kg/m^3$.

2.2 Mix Proportions

Mortar mixtures were made in a 1:4 ratio. The paste (cement + water) content was decreased using the paste replacement method. 10%, 20%, and 30% of the paste was replaced with marble waste powder finer than 90 μm. For the study, water to cement ratios of 0.7, 0.8, and 0.9 and a curing period of 7, 28, and 90-day was chosen.

2.3 Design of Experiments

The Taguchi method was used to systematically look at how different factors affect the compressive strength of mortar and to find the best combination of process parameters. L9 orthogonal array is used to cut down on the number of experiments while still getting the main effects of each parameter. The three chosen factors were:

- A – Replacement Level (R/L) (%): 10%, 20%, 30%
- B – Water-Cement Ratio (W/C): 0.7, 0.8, 0.9
- C – Curing Period (C/P) (days): 7, 14, 28

2.4 Optimisation of Parameters

Compressive strength (MPa) was used as the criterion for comparison when assessing the performance of each trial. A comparison of the Signal-to-Noise (S/N) ratio and the mean response values was carried out in order to make optimization simpler to understand. For the purpose of determining S/N ratios, the "larger-is-better" criterion was utilized considering that the targeted objective was to achieve a higher compressive strength. For the S/N ratio, the formula is as follows:

Where y_i is the response value for each trial and n is the number of repetitions. A response table was created for both the means and signal-to-noise ratios, enabling the identification of the most significant parameter levels. The level exhibiting the highest average signal-to-noise ratio and mean value for each

$$\text{S/N Ratio} = -10 \cdot \log_{10}\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right)$$

factor was considered ideal.

2.5 Influence of Process Parameters

An Analysis of Variance (ANOVA) was conducted to evaluate the statistical significance and relative contribution of each process parameter on the compressive strength of cement mortar including marble waste. ANOVA is a statistical method employed to assess the impact of independent factors on a dependent variable by analysing the variability both within and among experimental groups. Each factor (R/L, W/C, C/P) was assessed at three levels according to the L9 Taguchi design. The ANOVA was performed utilising the Adjusted Sum of Squares (Adj SS) for each parameter, and the F-value was employed to evaluate the factor's impact in relation to the error variance. The P-value denotes statistical significance within a 95% confidence interval.

2.6 Microstructural Characterization (SEM)

Fractured surfaces of mortar specimens were subjected to scanning electron microscopy (SEM) investigation following a cure period of 28 days. A gold coating was applied to the samples before they were analysed, and they were examined at different magnifications in order to evaluate morphological characteristics such as the production of C–S–H gels, ettringite, $Ca(OH)_2$ crystals, and porosity.

2.7 Elemental Composition (EDAX)

Energy Dispersive X-ray Analysis (EDAX) and Scanning Electron Microscopy (SEM) was used on the same areas to figure out what kinds of oxides were in the mortar samples that had marble debris in them. This method lets us to know qualitatively and semi-quantitatively how elements such as such as CaO, SiO_2, Al_2O_3, Fe_2O_3, SO_3, and alkalis (Na_2O and K_2O) are spread out in the microstructure. This gives us information on hydration products and the presence of unreacted or crystalline phases.

3. RESULTS AND DISCUSSION

3.1Fresh Properties

MW integrated mortar mixes were assessed for workability by the flow table test and cone penetrometer test at replacement levels of 5, 10, 15, 20, 25, 30, 40, and 50%, utilising water-cement ratios of 0.7, 0.8, and 0.9. Figure 1 demonstrates the workability of MW, indicating a variation in horizontal flow from 107 to 141 mm and a vertical penetration depth measuring the cohesion ranging from 4 to 28 mm. As the percentage of marble waste replacement grows from 10% to 50%, both flow value and cone penetration diminish, signifying a gradual decline in workability and an increase in stiffness with elevated marble waste content. The decrease is more pronounced at a lower water-to-cement ratio (0.7) and more gradual at a higher ratio (0.9), indicating that the inclusion of extra mixing water partially mitigates the water consumption induced by the fineness of marble debris. Beyond 30% replacement, the curves start to converge, particularly in cone penetration, indicating a diminishing advantage of more water once fines predominate rheology.

3.2Optimisation of Parameters

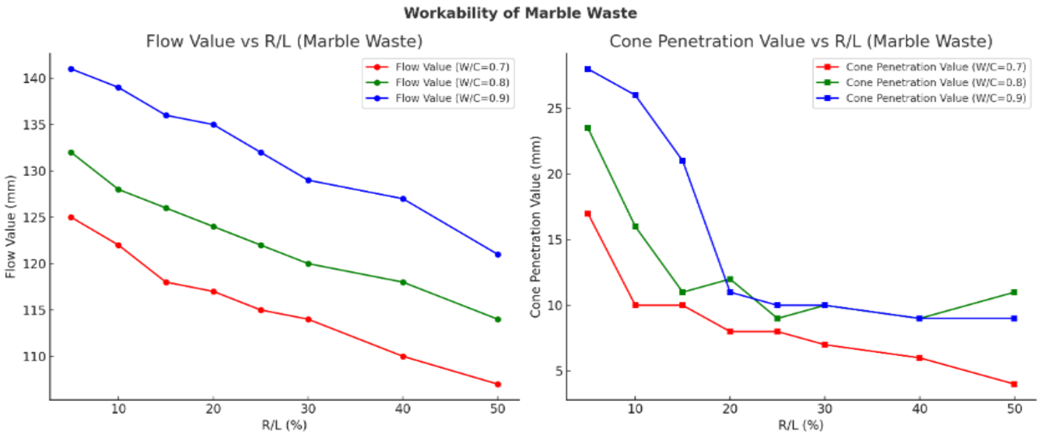

Figure: 1 Workability of Marble Waste Mortar Mixes by Flow Table Test and Cone Penetration Test

3.2.1 By Signal to Noise Ratio

Table 1 and Figure 2 indicate that the Replacement Level (R/L) exhibits the highest delta (5.42), signifying its predominant influence on compressive strength according to the S/N ratio. This is succeeded by the Curing Period (C/P) with a delta of 4.47, while the water to cement ratio (W/C) demonstrates the minimal impact with a delta of 0.58. The signal-to-noise ratios diminish as replacement levels increase, signifying that elevated replacement levels result in reduced strength. Conversely, extended curing durations markedly enhance strength, as seen by the upward trend observed from 7 to 90 days.

Table:1 Response for Signal to Noise Ratio

Response Table for Signal to Noise Ratios			
Larger is better			
Level	R/L	W/C	C/P
1	27.13	24.34	22.04
2	25.19	24.78	25.47
3	21.71	24.91	26.51
Delta	5.42	0.58	4.47
Rank	1	3	2

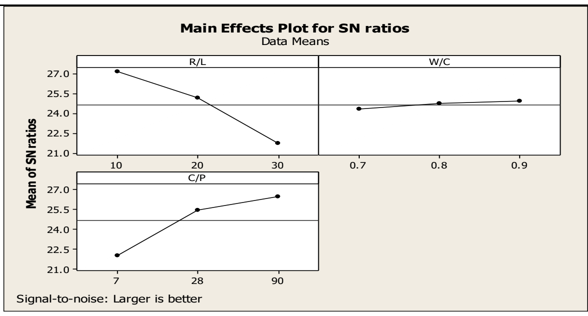

Figure:2 Main Effect Plot for Signal to Noise Ratio

3.2.2 By Means

Table 2 and Figure 3 indicate that the Replacement Level (R/L) exhibits the highest delta 11.67, signifying its predominant influence on compressive strength according to the Means optimisation. This is succeeded by the Curing Period (C/P) with a delta of 9.08, while the water to cement ratio (W/C) demonstrates the minimal impact with a delta of 2.5. The means value diminishes as replacement levels increase, signifying that elevated replacement levels result in reduced strength. Conversely, extended curing durations markedly enhance strength, as seen by the upward trend observed from 7 to 90 days similar to that obtained by S/N ratio.

Table :2 Response for Means

Response Table for Means			
Level	R/L	W/C	C/P
1	24.13	16.7	12.89
2	18.31	19	20.02
3	12.46	19.2	21.98
Delta	11.67	2.5	9.08
Rank	1	3	2

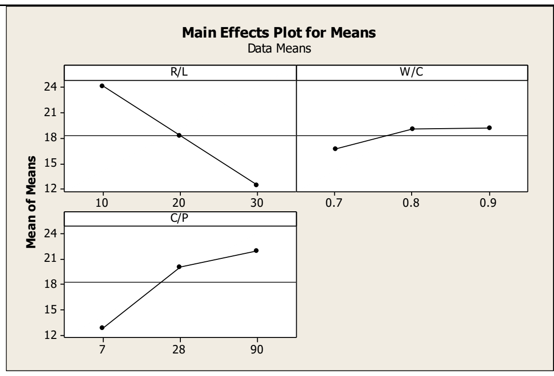

Figure:3 Main Effect Plot for Means

3.3Influence of Process Parameter

Table 3 and Figure 4 illustrate the impact of the parameters R/L, W/C, and C/P. Replacement Level (R/L) accounts for the majority (204.19/409.53 = 49.9%), signifying that marble waste content exerts the most significant impact on compressive strength. The Curing Period (C/P) constitutes 33.5%, indicating a substantial impact, as extended curing enhances hydration and strength progression. The Water-Cement Ratio (W/C) exerts a negligible effect (~2.8%), suggesting its influence is comparatively minor within the examined range. All P-values exceed 0.05, signifying that none of the factors are statistically significant at the 95% confidence level. This may result from the limited number of replications (few degrees of freedom). S = 5.32 MPa signifies the standard deviation of the measured values from the regression line. A reduced value would signify enhanced forecast accuracy. This score indicates a considerable degree of variability in the experimental results. The model explains a significant portion of the variance in compressive strength, as indicated by R^2 = 86.17%.

Table:3 Anova for Compressive Strength

Analysis of Variance for COMP STR, using Adjusted SS for Tests						
Source	**DF**	**Seq SS**	**Adj SS**	**Adj MS**	**F**	**P**
R/L	2	204.19	204.19	102.1	3.61	0.217
W/C	2	11.55	11.55	5.78	0.2	0.831
C/P	2	137.17	137.17	68.58	2.42	0.292
Error	2	56.62	56.62	28.31		
Total	8	409.53				
S = 5.32060 R-Sq = 86.17% R-Sq(adj) = 44.70%						

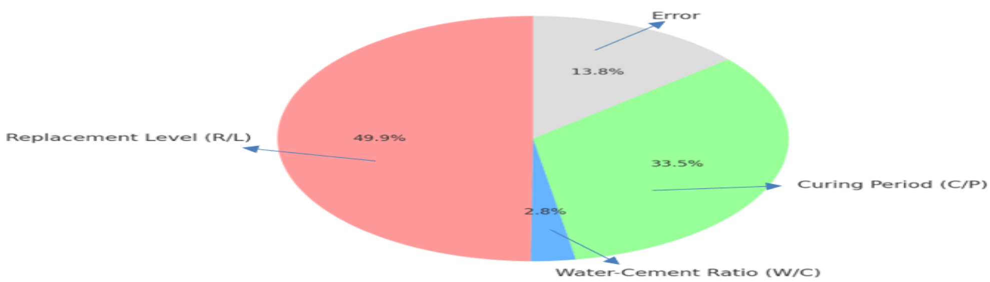

Figure: 4 Effect of Process Parameters on Compressive Strength

3.4 SEM Analysis

SEM micrographs (figure:5) revealed that the 10% replacement microstructure shows a matrix that is well-compacted and dense, with hydration products that are easy to see. The presence of Calcium Silicate Hydrate (C–S–H) gel, Calcium Hydroxide ($Ca(OH)_2$) platelets, and needle-like ettringite crystals shows that the hydration process is moving forward. The structure has low porosity and high particle interlocking, which means that marble debris at this level works well as a filler, improving the microstructure and helping to build strength. At 20% replacement, the sample has a denser and more fibrous matrix, and there are flocculated hydration products in it. The shape suggests that C–S–H gel and maybe ettringite crystals are still forming. The higher packing density and fewer voids at this level show that marble waste is still helpful by refining the pore structure. At 30% replacement the microstructure becomes more porous, and bigger voids and microcracks start to show up. These characteristics suggest that the material is not getting enough water, which could be because it has too much inert substance or not enough effective binder. The fact that the matrix doesn't hold together well and that there are un hydrated or loosely linked particles.

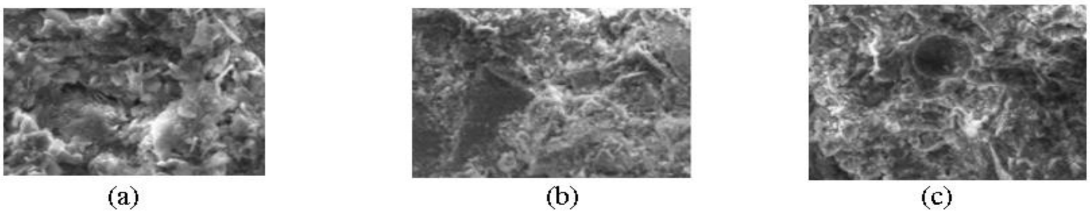

Figure 5: SEM Image –(a) 10% (b) 20% (c) 30% Replacement

3.5 EDAX Analysis

EDAX results (table:4) showed high CaO and SiO_2 concentrations, which validate the existence of crucial cementitious phases and the successful integration of marble debris into the matrix. Al_2O_3 and SO_3 concentrations facilitate the development of secondary hydration products such as ettringite. The presence of alkalis (Na_2O, K_2O) is negligible and unlikely to negatively impact performance. The comprehensive oxide profile indicates that marble debris serves not only as a filler but also partially enhances the hydration process through its reactive properties.

Table: 4 Oxide Composition of the Mixes With –10%, 20%, 30% Replacement

Oxide	10%	20%	30%
Na_2O	1.60	1.05	1.05
MgO	1.46	1.33	0.74
Al_2O_3	9.31	8.13	8.34
SiO_2	35.46	37.57	48.28
SO_3	1.44	1.22	2.28
K_2O	1.49	1.55	1.93
CaO	46.65	46.01	35.27
Fe_2O_3	2.58	3.13	2.11

3.4Comparative Microstructural Evaluation

SEM and EDAX analyses reveal a clear transition in microstructure with increasing marble waste content. At 10% replacement, the microstructure is dominated by hydration, leading to strength enhancement through densification and filler effects. However, at 30% replacement, reduced hydration and increased porosity result in a weaker microstructure and lower mechanical performance. The increased presence of silica and sulfates supports the formation of C–S–H gel and ettringite, contributing to strength development. Overall, marble waste shows considerable potential to enhance mortar properties through both physical and chemical mechanisms.

4. CONCLUSIONS

The incorporation of marble waste into cement mortar has emerged as a promising strategy for enhancing sustainability in construction without significantly compromising performance. Throughout this study, the influence of varying MW replacement levels on the physical, mechanical, and microstructural properties of MM5 grade mortar was systematically evaluated. Experimental observations, supported by SEM and EDAX analyses, offered insights into the evolving morphology and chemical composition of the mortar with increasing MW content. Workability, hydration behaviour, and pozzolanic activity were examined alongside statistical modeling to understand the reliability and significance of the findings. The following results point to an optimal range of MW substitution that balances environmental benefits, mechanical performance, and practical usability.

1. Marble waste significantly enhances the hydration and densification of cement mortar when substituted at levels up to 30%, achieving a typical strength of 5-7 MPa for MM5 grade mortar.
2. SEM examination indicated an enhanced development of C–S–H gel and ettringite at 20% replacement levels, resulting in decreased porosity.
3. EDAX verified increasing amounts of SiO_2 and SO_3, which augment pozzolanic activity and facilitate microstructural growth.
4. Higher MW makes it harder to work with, which can trap air and make it harder to spread, which is what the SEM shows at 30% replacement with bigger voids and loosely bonded areas.
5. A better flow at 10% MW helps with good packing and dispersion, which is in line with the compact C–S–H matrix and low porosity seen under a microscope.
6. All parameters exhibited P-values greater than 0.05, indicating that none of the factors are statistically significant at the 95% confidence level. The practical importance of R/L and C/P is evident due to their considerable percentage contributions.
7. The significant disparity between R^2 and R^2(adj) suggests that certain variability accounted for by the model may lack significance and could result from random fluctuation.
8. For eco-friendly mixtures that want to use less cement, 10–20% MW is a good equilibrium because it saves cement while still being easy to work with.
9. At ≥30% MW, need for chemical admixtures prevails to improve workability, or rheology modifiers to get the placement performance back.
10. Marble waste serves as a sustainable alternative binder, enhancing microstructural integrity and long-term durability.

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