Investigation of Properties of Mortars Containing Waste Stone Powder Instead of Sand Under Freezing-Thawing Effect

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Abstract. Nowadays, sustainable production is vitally important in consideration of limited raw materials and an environmental welfare. Sustainable production encourages the use of several industrial wastes as raw materials in different fields. Water and aggregate are among the most consumed items on the earth. Waste powders, with a size of 75 μm or less, formed during the aggregate production process affect both the environment and human health negatively. For sustainable aggregate production, it is important that the waste powder generated during the aggregate production phase is used in the concrete industry. Limestone, basalt and dolomite are commonly used as concrete aggregate. In this study, limestone, basalt and dolomite waste powders were replaced with fine aggregate (sand). Reference samples (0 %) and 20% -30% waste stone powder produced by replacing standard sand with mortar. The freeze-thaw effect (40 cycles) on compressive strength, flexural strength, ultrasonic pulse velocity, weight loss are analysed. For this purpose, a total of 105 (21×5) 40×40×160 mm prism samples were produced. Under the influence of freeze-thaw cycles, the loss of weight in samples produced by replacement of standard sand and waste stone powders is less than that of the reference samples. The ultrasonic pulse velocity tend to increase in general as the number of freeze-thaw cycles increases. The flexural strength rises as the replacement rate is increased, and after the freeze-thaw cycle, a slight decline is observed. The compressive strength values decrease as the replacement rate is increased. It decreases at the end of the 10th freeze-thaw cycle but the variability tend decrease at extending cycles (20-40).

1. Introduction
In sustainable production, the usability of waste products materials as a raw materials in different production areas is important. The re-use of the waste aggregate and washing water that generated during the concrete production is realized in concrete plants [1], [2]. Aggregates are constitute about 60-70% of concrete. For this reason, the re-use of waste powder materials that are obtained in aggregate quarry is required for the sustainable concrete. There are many studies within the usability of waste powder materials in concrete production.

The effect of quartz dust, marble dust and crushed stone aggregates on the compressive strength of concrete are investigated [3-6]. Limestone powders have positive effects on self-compacting concrete properties [7-8].
The physical, mechanical and durability properties of the mortar samples containing waste basalt powder (obtained in Tekirdağ) (replacement with the fine aggregate and cement) have been investigated. The usability of the waste basalt powders in mortars have been investigated and the use rate of the powder in concrete production tried to determine [9]. The effects of the different filler materials on physical, mechanical and microstructure of self-compacting and flow-able concrete were investigated for two groups of filler as pozzolanic filler (silica fume and metakaolin) and non-pozzolanic filler (limestone powder, granite dust, and marble dust) [10].

Generally, the effects of waste powder materials on physical and mechanical properties of concrete or mortar are examined. And also the effect of the use of marble powder on mortar properties have been investigated under freeze-thaw effect [11]. The weight loss, flexural strength and compressive strength of mortars containing waste powder (limestone, basalt and dolomite) that replacement with standard sand (%5, %10) have been investigated under freeze-thaw effect (10, 20, 30 and 40 cycles) [12].

In this study, the mortar samples containing different powder materials (limestone, basalt and dolomite) that replacement (%20 and %30) to standard sand were produced. The weight loss, water absorption, ultrasonic pulse velocity, flexural strength and compressive strength of the mortar samples containing different waste powder (limestone, basalt and dolomite) were determined under freezing-thawing effect (10, 20, 30 and 40 cycles).

2. Materials and methods
In this study, the mortar containing limestone, basalt and dolomite powders that replacement with the standard sand (%20, %30) are produced. Chemical composition of constituent materials of mortar are given in Table 1 and physical and mechanic properties of is given in Table 2. The mix proportion of mortar prepared for this study is given in Table 3. Melamine sulphonate polymer based superplasticizer was used at varying dosages for the workability. Drinking water was used for production of cement mortar.

| Compositions (%) | CEM I 42.5 R | Limestone powder | Basalt powder | Dolomite powder |
|-----------------|--------------|------------------|--------------|----------------|
| CaO             | 62.85        | 54.65            | 9.01         | 11.69          |
| SiO₂            | 19.52        | 1.55             | 42.82        | 41.70          |
| Al₂O₃           | 5.36         | 0.71             | 13.68        | 13.67          |
| Fe₂O₃           | 3.38         | 0.26             | 10.25        | 9.12           |
| MgO             | 1.15         | 0.27             | 10.33        | 8.39           |
| SO₃             | 3.36         | 0.024            | 0.053        | 0.19           |
| Na₂O/K₂O        | 0.22/0.72    | 0.016/0.088      | 2.73/1.87    | 2.64/1.54      |
| Cl               | 0.0415       | -                | -            | -              |
| H₂O             | -            | -                | -            | -              |
| Loss of ignition | 3.29         | 42.37            | 5.6          | 7.44           |
| Insoluble residue | 0.24        | -                | -            | -              |

2.1. Test Setup and Procedure
Cement mortars were produced according to TS EN 196-1 [13]. The consistency of fresh mortar was determined by using the flow table. Test specimens were cured further in water.

Compressive and flexural strength tests according to TS EN 196-1 [13], ultrasonic pulse velocity tests according to TS EN 12504-4 [14] was performed on prism specimens with dimensions of 40×40×160 mm. 3 specimens were tested each cycles for reference mixture and three specimens were tested each cycles for other mixtures, and average was reported in this paper. Totally, 105 prism
specimens were produced. All the samples were coded in the form of BS20 (the meaning of first word is waste powder (Ref, reference, B, basalt, L, limestone and D, dolomite) and second word is replacement materials (sand), and the meaning of the number is replacement ratio (%20 and %30). Freeze-thaw tests was performed according to ASTM C-666 [15] and 40 cycles was realized.

3. Results and discussions

Production was carried out with 20% and 30% replacement of CEN standard sand with limestone, basalt and dolomite powders. The samples were subjected to 10, 20, 30 and 40 cycles of freeze-thaw effect. In these cycles, samples, compression strength, flexural strength, ultrasonic pulse velocity and weight loss values were determined. Depending on the number of cycles, the change in weight loss, ultrasonic pulse velocity, compressive strength and flexural strength are shown in Figure 1, Figure 2, Figure 3 and Figure 4, respectively.
Figure 1. Variations of weight loss to the number of cycles.

Figure 2. Variations of ultrasonic pulse velocity to the number of cycles.

Figure 3. Variations of compressive strength to the number of cycles.
Figure 4. Variations of flexural strength to the number of cycles.

Weight loss is increased as the number of cycles increasing in reference samples. Generally, as the number of cycles increase, the weight loss variation is so little in the mortar samples containing waste powder. However, weight loss was increased in the LS30 sample when the freeze-thaw cycles.

As can be seen Figure 2, generally, as the number of cycles increases, the ultrasonic pulse velocity increases.

As can be seen in Figure 3, the compressive strength is decreased with the replacement ratio. Especially, decreasing of the compressive strength is high at the end of the 10 cycles.

Any variation is not observed in the flexural strength to the end of the 20 cycles. However, a significant decrease is observed at the end of the 30 and 40 cycles. Generally, the flexural strength is increased with the increasing of the replacement ratio.

The results of the analysis of variance and the statistical analysis of the compression strength, flexural strength and weight loss are given in Tables 4 and Table 5, respectively.

### Table 4. Analysis of variance.

| Source                        | Sum of squares | df  | Mean square | F Value | p-value Prob > F | Significant level |
|-------------------------------|----------------|-----|-------------|---------|------------------|-------------------|
| ANOVA for reduced cubic model-Weight loss | 0.5341 | 15  | 0.0356      | 53.70   | < 0.0001         | significant       |
| A-Replacement ratio           | 0.2928 | 1   | 0.2928      | 441.53  | < 0.0001         |                   |
| B-Number of cycles           | 0.0058 | 1   | 0.0058      | 8.70    | 0.0079           |                   |
| C-Powder                     | 0.0387 | 2   | 0.0193      | 29.17   | < 0.0001         |                   |
| AB                            | 0.0355 | 1   | 0.0355      | 53.62   | < 0.0001         |                   |
| AC                            | 0.0538 | 2   | 0.0269      | 40.60   | < 0.0001         |                   |
| BC                            | 0.0098 | 2   | 0.0049      | 7.38    | 0.0040           |                   |
| A²                            | 0.0105 | 1   | 0.0105      | 15.85   | 0.0007           |                   |
| ABC                           | 0.0073 | 2   | 0.0036      | 5.49    | 0.0125           |                   |
| A²B                           | 0.0183 | 1   | 0.0183      | 27.65   | < 0.0001         |                   |
| A²C                           | 0.0616 | 2   | 0.0308      | 46.46   | < 0.0001         |                   |
| Residual                      | 0.0133 | 20  | 0.0007      |         |                  |                   |
| Corrected Total              | 0.5474 | 35  |             |         |                  |                   |
Table 5. Analysis of variance (continue).

| Source                        | Sum of squares | df | Mean square | F Value | p-value | Significant level |
|-------------------------------|----------------|----|-------------|---------|---------|-------------------|
| ANOVA for reduced cubic model-Ultrasound pulse velocity |               |    |             |         |         |                   |
| Model                         | 0.0991         | 11 | 0.0090      | 11.28   | <0.0001 | significant       |
| A- Replacement ratio          | 0.0012         | 1  | 0.0012      | 1.47    | 0.2335  |                   |
| B- Number of cycles           | 0.0481         | 1  | 0.0481      | 60.24   | <0.0001 |                   |
| C- Powder                     | 0.0055         | 2  | 0.0028      | 3.47    | 0.0429  |                   |
| AB                            | 0.0011         | 1  | 0.0011      | 1.37    | 0.2503  |                   |
| BC                            | 0.0072         | 2  | 0.0036      | 4.52    | 0.0183  |                   |
| A²                            | 0.0007         | 1  | 0.0007      | 0.8671  | 0.3585  |                   |
| B²                            | 0.0098         | 1  | 0.0098      | 12.27   | 0.0013  |                   |
| AB²                           | 0.0068         | 1  | 0.0068      | 8.46    | 0.0065  |                   |
| AB                            | 0.0187         | 1  | 0.0187      | 23.45   | <0.0001 |                   |
| Residual                      | 0.0264         | 33 | 0.0008      |         |         |                   |
| Corrected Total               | 0.1255         | 44 |             |         |         |                   |
| ANOVA for reduced cubic model-Compressive strength |               |    |             |         |         |                   |
| Model                         | 636.12         | 6  | 106.02      | 7.13    | <0.0001 | significant       |
| A- Replacement ratio          | 0.0801         | 1  | 0.0801      | 0.0054  | 0.9419  |                   |
| B- Number of cycles           | 229.24         | 1  | 229.24      | 15.41   | 0.0004  |                   |
| AB                            | 141.81         | 1  | 141.81      | 9.53    | 0.0038  |                   |
| A²                            | 99.31          | 1  | 99.31       | 6.68    | 0.0137  |                   |
| B²                            | 116.78         | 1  | 116.78      | 7.85    | 0.0079  |                   |
| AB²                           | 61.49          | 1  | 61.49       | 4.13    | 0.0491  |                   |
| Residual                      | 565.24         | 38 | 14.87       |         |         |                   |
| Corrected Total               | 1201.36        | 44 |             |         |         |                   |
| ANOVA for reduced cubic model-Flexural strength |               |    |             |         |         |                   |
| Model                         | 64.21          | 11 | 5.84        | 21.69   | <0.0001 | significant       |
| A- Replacement ratio          | 22.64          | 1  | 22.64       | 84.13   | <0.0001 |                   |
| B- Number of cycles           | 14.65          | 1  | 14.65       | 54.44   | <0.0001 |                   |
| C- Powder                     | 0.6736         | 2  | 0.3368      | 1.25    | 0.2993  |                   |
| AB                            | 10.08          | 1  | 10.08       | 37.44   | <0.0001 |                   |
| BC                            | 2.79           | 2  | 1.39        | 5.17    | 0.0111  |                   |
| A²                            | 0.9810         | 1  | 0.9810      | 3.65    | 0.0650  |                   |
| B²                            | 6.87           | 1  | 6.87        | 25.51   | <0.0001 |                   |
| AB²                           | 1.68           | 1  | 1.68        | 6.25    | 0.0176  |                   |
| Residual                      | 8.88           | 33 | 0.2691      |         |         |                   |
| Corrected Total               | 73.09          | 44 |             |         |         |                   |

Table 6. Statistical analysis.

|                      | Standard Deviation | Mean | R-Squared | Adjusted R-Squared | Prediction R-Squared | Adequate Precision |
|----------------------|--------------------|------|-----------|--------------------|----------------------|--------------------|
| Weight loss, %       | 0.0257             | -0.1767 | 0.9758   | 0.9576              | 0.9095               | 30,2071            |
| Ultrasonic pulse velocity, km/s | 0.0283 | 4.38 | 0.7900 | 0.7200 | 0.5366 | 15,8192 |
| Compressive strength, MPa | 3.86    | 38.26 | 0.5295 | 0.4552 | 0.3358 | 9,7255 |
| Flexural strength, MPa | 0.5188 | 8.5  | 0.8785 | 0.8380 | 0.7304 | 19,6470 |

As can be seen in Table 4, the main terms A, B and C (p-value, <0.0001, 0.0079, <0.0001, respectively), two factor interaction terms AB, AC and BC (p-value, <0.0001, <0.0001, 0.0040, respectively), quadratic term A² (p-value, 0.0007) and ABC, A²B, A²C (p-value, 0.0125, <0.0001,
<0.0001, respectively) terms have a significant effect on weight loss. Regression coefficients of weight loss have a similar value (0.9758-0.9576-0.9095).

The main terms B and C (p-value, <0.0001, 0.0429, respectively), two factor interaction term BC (p-value, 0.0183), quadratic term B² (p-value, 0.0013), A²B and AB² terms (p-value, 0.0065, <0.0001, respectively) have a significant effect on ultrasonic pulse velocity. A, AB and A² terms (p-value, 0.2335, 0.2503, 0.3585, respectively) do not have a significant effect on ultrasonic pulse velocity. These terms are included in the model because of the hierarchical model. Regression coefficients of ultrasonic pulse velocity is in reasonable agreement with each other (the difference between the adjusted-R² and prediction R² is less than 0.2) (0.7900, 0.7200 and 0.5366).

B, AB, A², B² and AB² terms have a significant effect (p-value, 0.0004, 0.0038, 0.0137, 0.0079, 0.04910, respectively) on compressive strength. The main term A is included in the model for hierarchical model. Regression coefficients of compressive strength show the appropriate variability (0.5295, 0.4552 and 0.3358).

A, B, AB, BC, A², B², A²B and AB² terms have a significant effect (p-value, <0.0001, <0.0001, <0.0001, 0.0111, 0.0650, <0.0001, 0.0176, 0.0006, respectively) on flexural strength. The main term C is included in the model for hierarchical model. The regression coefficients of flexural strength have a similar value (0.8785-0.8380-0.7304).

Final equations in terms of actual factor for weight loss (Y₁), ultrasonic pulse velocity (Y₂) and flexural strength (Y₃) are given in following Equations to the three different waste powder (basalt, limestone, dolomite), respectively.

**Powder (C) = Basalt,**

\[
Y_1 = -0.1627 - 0.0009 \cdot A - 0.0057 \cdot B + 0.0008 \cdot AB + 0.0001 \cdot A^2 - 0.00002 \cdot A^2B \]  \hspace{1cm} (1)

\[
Y_2 = 4.2534 + 0.0084 \cdot A + 0.0124 \cdot B - 0.0006 \cdot AB - 0.0002 \cdot A^2 - 0.0003 \cdot B^2 + 9.3586 \times 10^{-6} \cdot A^2B + 9.7762 \times 10^{-6} \cdot AB^2 \]  \hspace{1cm} (2)

\[
Y_3 = 8.4115 + 0.1612 \cdot A + 0.0961 \cdot B - 0.0071 \cdot AB - 0.0045 \cdot A^2 - 0.0047 \cdot B^2 + 0.0001 \cdot A^2B + 0.0001 \cdot AB^2 \]  \hspace{1cm} (3)

**Powder (C) = Limestone,**

\[
Y_1 = -0.1511 - 0.0259 \cdot A - 0.0062 \cdot B + 0.0010 \cdot AB + 0.0010 \cdot A^2 - 0.00002 \cdot A^2B \]  \hspace{1cm} (4)

\[
Y_2 = 4.3026 + 0.0084 \cdot A + 0.0111 \cdot B - 0.0006 \cdot AB - 0.0002 \cdot A^2 - 0.0003 \cdot B^2 + 9.3586 \times 10^{-6} \cdot A^2B + 9.7762 \times 10^{-6} \cdot AB^2 \]  \hspace{1cm} (5)

\[
Y_3 = 8.2171 + 0.1612 \cdot A + 0.0952 \cdot B - 0.0071 \cdot AB - 0.0045 \cdot A^2 - 0.0047 \cdot B^2 + 0.0001 \cdot A^2B + 0.0001 \cdot AB^2 \]  \hspace{1cm} (6)

**Powder (C) = Dolomite,**

\[
Y_1 = -0.1649 + 0.0006 \cdot A - 0.0056 \cdot B + 0.0008 \cdot AB + 6.7798 \times 10^{-6} \cdot A^2 - 0.00002 \cdot A^2B \]  \hspace{1cm} (7)

\[
Y_2 = 4.2596 + 0.0084 \cdot A + 0.0133 \cdot B - 0.0006 \cdot AB - 0.0002 \cdot A^2 - 0.0003 \cdot B^2 + 9.3586 \times 10^{-6} \cdot A^2B + 9.7762 \times 10^{-6} \cdot AB^2 \]  \hspace{1cm} (8)
\[ Y_3 = 7.7515 + 0.1612 \cdot A + 0.1329 \cdot B - 0.0071 \cdot AB - 0.0045 \cdot A^2 - 0.0047 \cdot B^2 + 0.0001 \cdot A^2B + 0.0001 \cdot A \cdot B^2 \] (9)

Final equations in terms of actual factor for compressive strength \((Y_4)\) is given in Equation 10.

\[ Y_4 = 48.8292 + 0.0614 \cdot A - 1.0366 \cdot B + 0.0325 \cdot AB - 0.0160 \cdot A^2 + 0.0181 \cdot B^2 - 0.00002 \cdot A^2B \] (10)

The contours and 3D plots of the two-level terms are given in following Figures.

Figure 5. Contour and 3D plots of AB term for weight loss.
Figure 6. Contour and 3D plots of AB term for weight loss (continue).

Figure 7. Contour and 3D plots of AB term for ultrasonic pulse velocity.
Figure 8. Contour and 3D plots of AB term for ultrasonic pulse velocity (continue).

Figure 9. Contour and 3D plots of AB term for flexural strength.
Figure 10. Contour and 3D plots of AB term for flexural strength (continue).

Figure 11. Contour and 3D plots of AB term for compressive strength.
Figure 12. Contour and 3D plots of AB term for compressive strength (continue).

4. Conclusions
The usability of the waste powder materials (limestone, basalt and dolomite) in construction industry as a raw material in the concrete or mortar by replacement with the CEN standard sand is important for sustainable production.

In this study, effect level of the waste powder type (categorical factor), replacement ratio (numerical factor) and number of cycle (numerical factor) on the weight loss, ultrasonic pulse velocity, compressive strength and flexural strength properties of mortar samples were determined and the usability of the waste powder materials in concrete production was investigated. The results suggest that;

- As the replacement ratio increased, weight loss and compressive strength are decreased but ultrasonic pulse velocity and flexural strength are generally increased.

- As the number of cycles increased, weight loss is increased for LS30 and Ref samples but for the other samples weight loss is stable.

- As the number of cycles increased, ultrasonic pulse velocity is increased but compressive strength and flexural strength are decreased.

- A, B, C, AB, AC, BC, A², ABC and A²B terms have a significant effect on the weight loss.

- B, C, BC, B², A²B and AB² terms have a significant effect on the ultrasonic pulse velocity.

- B, AB, A², B² and AB² terms have a significant effect on the compressive strength.

- A, B, AB, BC, A², B², A²B and AB² terms have a significant effect on the flexural strength.

The fact that waste powder types have no significant effect on compressive strength and flexural strength is due to the similar fineness of the waste powder materials.

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