Correlation of Ultrasound Pulse Velocity with Pozzolanic Activity and Mechanical Properties in Lime-Calcined Clay Mortars

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ABSTRACT

The relationship between ultrasound pulse velocity (UPV) with pozzolanic activity of lime mortars produced with clays calcined at different temperatures were examined. Three different types of clay samples were calcined at 550, 650, 750 and 850 °C. All the calcined clays were ground in proper granulometric sizes and with them, a series of 12 batches of lime mortars were mixed. From three per mix, a total of 36 samples were prepared in accordance with TS 25 standard. Two extra batches of mortars were prepared for comparison purposes; pure lime mortar and a pozzolanic lime mortar mixed with rock powder. All the samples were cured in accordance with ASTM C593-95 standard. There is a close and good relationship between the UPV with pozzolanic activity and mechanical properties of lime mortars produced with clays calcined at different temperatures. Hence, pozzolanic activity can be predicted also by non-destructive methods.

Keywords: Lime/Clay Mortar; Calcination; Ultrasound Pulse Velocity; Pozzolanic Activity; Mechanical Properties

1. INTRODUCTION

Analyzing the properties of materials and establishing relationships between them through non-destructive tests is an active area of study and many different methods were developed for this purpose. One of these is the measurement of ultrasound pulse velocity (UPV) which provides important information about the internal structure of materials. With this technique, relationships between internal structure, mechanical properties and dynamic modulus of elasticity (E_d) of numerous materials can be studied. UPV is commonly used to predict and evaluate the mechanical properties of various materials in a non-destructive way [1]. Many studies were made on the relationships between UPV and strength and, elastic properties of natural [2] and artificial stones, especially those of cement-binder concrete and mortars [3-8] as well as wood materials [9, 10]. Studies on checking pozzolanic activity with UPV and evaluating the relationship between the two are inadequate in the literature. Consequently, this study addressed the relations of UPV with pozzolanic activity and strength properties of lime mortars produced with calcined clays.

Propagation speed of sound waves within materials changes in accordance with the type and inner characteristics of the material. Due to this behavior, information about the inner structure of the material can be obtained. A high transmission rate of sound means less pores and, thus higher strength. When the pulse is transferred to the material by a transducer, it undergoes multiple reflections at the boundaries of different phases...
within the material. A complex system of stress waves is developed which includes both longitudinal and shear waves propagating through the material. The pulse velocity \( V \) (in km/s or m/s) is given by:

\[
V = \frac{l}{t}
\]

where \( l \) is the path length and \( t \) the time it takes the pulse to travel this distance. The pulse velocity, \( (V) \), of longitudinal stress waves in a material is related to its elastic properties and density according to following relationship:

\[
E_d = 10^7 \cdot V^2 \cdot \Delta / g
\]

where \( (E_d) \) (kN/mm\(^2\)) is the dynamic modulus of elasticity, \( (V) \) (\(\mu/s\)) the pulse velocity, \( (\Delta) \) the density of material (g/cm\(^3\)) and \( (g) \) (9.8 m/s\(^2\)) the gravitational acceleration \[11\].

By conducting in-field tests on historical and modern structures important information about the state of the materials in the structure can be obtained. Many structural properties such as microstructure, distribution of micro cracks, the quality of bonding at interfaces between different components forming the material and rheological and mechanical properties of components can be evaluated by ultrasonic methods. The ultrasonic method was used to predict the hydration activity of fly ash cement binder composites \[7\]. The velocity of ultrasonic waves is sensitive to the additives and admixtures accelerating the setting and hardening behavior of concrete and mortar \[12\]. Ultrasound may be used to characterize the mechanical properties of cement and epoxies as well as other materials \[13, 14\]. Because the speed and attenuation of sound waves are sensitive to the viscoelastic properties of the material, ultrasound can be used to monitor the curing process of cement composites \[7\]. Mineral admixtures increase the UPV value of concrete \[15\]. This method can also be used to track the reaction between a pozzolanic material and Ca(OH)\(_2\) in the cement and lime binder composites.

### 1.1. Pozzolanic activity

Mortars containing pozzolanic materials are superior to hydrated lime mortars in terms of durability and mechanical properties. There is a relationship between the pozzolanic activity of a material and the mechanical behavior of mortars produced with that material. Mortars prepared with high reactive pozzolans have higher long term strength. This property is closely related to the extent of silicate formation resulting from reactions between Ca(OH)\(_2\) and the reactive minerals (SiO\(_2\), Al\(_2\)O\(_3\)) in the pozzolan. The silicate formation mechanism can be summarized as follows: In the mixture, lime produces the basic solution and reacts with active silica and alumina in the pozzolan to form complex hydrated calcium alumina-silicates. The minerals partially dissolve and release silica and alumina. These minerals react with calcium and hydroxyl ions to produce calcium silicate/aluminate hydrates (CSH and CAH) \[16\]. Improvements observed in the mechanical properties of binder as a function of time explain the effect of these hydrates. In a previous study \[17\], pozzolanic reactions between calcined clay, hydrated lime and water were examined and it was observed that composites of hydrated tetra-calcium aluminate (C\(_4\)AH\(_6\)), hydrated tri-calcium aluminate (C\(_3\)AH\(_6\)), hydrated calcium aluminate (CAH) and gehlenite (C\(_2\)ASH\(_3\)) form and this process is facilitated by the basic state created by lime. This structure enables the mortar to gain mechanical strength faster. In addition to the natural substances like those basically composed of silicon, aluminum and ferric oxide, volcanic ashes, tuff, bauxite, artificial ones like ashes from thermal power plants, blast furnace slag, powder of calcined clay, ashes of some burnt plant products (such as rice husk, coconut shell) can also be used as pozzolanic materials \[18\]. Also, minerals such as feldspar and zeolites may have pozzolanic properties \[19\].

Numerous factors such as mineralogical and chemical composition of the pozzolan, extent of the amorphous phase, specific surface area, level of dehydroxylation and the amount of Ca(OH)\(_2\) in binder, mixing conditions and water/binder ratio have effects on pozzolanic activity \[20\]. Clays calcined at different temperatures are typical samples in which to observe the development of pozzolanic activity and the mechanism of silicate formation, because clays may have different pozzolanic properties depending on the calcination temperature. The structure of the mineral and hence the type of clay are of importance as well. The optimum pozzolanic activity can be obtained by kaolinite clays calcined at temperatures between 500/550 - 800/850 °C \[21-25\]. Consequently, clays calcined at different temperatures constitute good samples for observing pozzolanic development through UPV. Thus, for this experimental study, lime mortars were produced from materials obtained by calcinations of three different types of clays at four specified temperatures and after being cured in standard conditions, their UPV values were assessed and an evaluation was made based on the data that obtained from mechanical tests.

### 2. Experimental program

#### 2.1. Materials, properties and methods

For use in the experiments, three different types of clays (kaolinite), red clay (K1), pink clay (K2) and grey clay (K3), were obtained from the Beykoz region in Istanbul. Also, the powder of a volcanic rock (andesite) (SM) was also used in the study for comparison. The chemical compositions and trace element contents of the clays and the rock powder were identified by ICP-ES analysis (Inductively Coupled Plasma Emission Spectroscopy, ACME Analytical Labs.) (Table 1 and Table 2).
Table 1. Chemical composition and physical properties of clays and rock dust

<table>
<thead>
<tr>
<th>Oxides</th>
<th>SiO₂ (%)</th>
<th>Al₂O₃ (%)</th>
<th>Fe₂O₃ (%)</th>
<th>MgO (%)</th>
<th>CaO (%)</th>
<th>Na₂O (%)</th>
<th>K₂O (%)</th>
<th>TiO₂ (%)</th>
<th>P₂O₅ (%)</th>
<th>MnO (%)</th>
<th>Cr₂O₃ (%)</th>
<th>LOI (%)</th>
<th>Sum (%)</th>
<th>TOT/C (%)</th>
<th>TOT/S (%)</th>
<th>δ (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>K1</td>
<td>59.85</td>
<td>14.56</td>
<td>7.57</td>
<td>2.96</td>
<td>5.66</td>
<td>1.45</td>
<td>2.62</td>
<td>0.94</td>
<td>0.20</td>
<td>0.12</td>
<td>0.03</td>
<td>3.8</td>
<td>99.9</td>
<td>0.70</td>
<td>0.03</td>
<td>2.43</td>
</tr>
<tr>
<td>K2</td>
<td>60.66</td>
<td>16.14</td>
<td>5.25</td>
<td>1.44</td>
<td>3.50</td>
<td>2.07</td>
<td>4.27</td>
<td>0.81</td>
<td>0.16</td>
<td>0.13</td>
<td>0.01</td>
<td>5.3</td>
<td>99.9</td>
<td>&lt;0.02</td>
<td>2.61</td>
<td></td>
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<tr>
<td>K3</td>
<td>57.52</td>
<td>13.22</td>
<td>1.53</td>
<td>5.28</td>
<td>12.10</td>
<td>0.70</td>
<td>4.40</td>
<td>5.73</td>
<td>0.41</td>
<td>0.25</td>
<td>0.10</td>
<td>1.8</td>
<td>99.9</td>
<td>0.14</td>
<td>&lt;0.02</td>
<td>2.46</td>
</tr>
<tr>
<td>Andesite</td>
<td>59.88</td>
<td>17.76</td>
<td>4.33</td>
<td>1.36</td>
<td>3.69</td>
<td>4.40</td>
<td>5.73</td>
<td>0.41</td>
<td>0.25</td>
<td>0.10</td>
<td>0.00</td>
<td>1.8</td>
<td>99.9</td>
<td>0.14</td>
<td>&lt;0.02</td>
<td>2.49</td>
</tr>
</tbody>
</table>

Table 2. Trace element contents of samples

<table>
<thead>
<tr>
<th>Elements</th>
<th>Cu (ppm)</th>
<th>Ba (ppm)</th>
<th>Zn (ppm)</th>
<th>Ni (ppm)</th>
<th>Co (ppm)</th>
<th>Sr (ppm)</th>
<th>Zr (ppm)</th>
<th>Ce (ppm)</th>
<th>Y (ppm)</th>
<th>Nb (ppm)</th>
<th>Sc (ppm)</th>
<th>Ta (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>K1</td>
<td>378</td>
<td>436</td>
<td>158</td>
<td>124</td>
<td>&lt;20</td>
<td>211</td>
<td>184</td>
<td>65</td>
<td>27</td>
<td>19</td>
<td>17</td>
<td>&lt;20</td>
</tr>
<tr>
<td>K2</td>
<td>126</td>
<td>723</td>
<td>75</td>
<td>27</td>
<td>&lt;20</td>
<td>218</td>
<td>179</td>
<td>58</td>
<td>22</td>
<td>15</td>
<td>10</td>
<td>&lt;20</td>
</tr>
<tr>
<td>K3</td>
<td>146</td>
<td>366</td>
<td>133</td>
<td>64</td>
<td>&lt;20</td>
<td>238</td>
<td>194</td>
<td>93</td>
<td>31</td>
<td>15</td>
<td>13</td>
<td>&lt;20</td>
</tr>
<tr>
<td>Andesite</td>
<td>175</td>
<td>886</td>
<td>66</td>
<td>52</td>
<td>39</td>
<td>423</td>
<td>107</td>
<td>&lt;30</td>
<td>21</td>
<td>10</td>
<td>7</td>
<td>&lt;20</td>
</tr>
</tbody>
</table>

According to the ASTM C 618-03 Standard [26] calcined clays form Class N pozzolan and it is recommended that the total weight fraction of SiO₂, Al₂O₃ and Fe₂O₃ content of the substance to be used in pozzolanic activity experiments be at least 70%. As seen from the chemical analysis results, all samples used in the study satisfy this condition. Lime used as binder in mortars prepared for the study, was procured from a supplier and its physical and chemical properties are given in Table 3. Standard sand, as specified in TS EN Standard 196-1 [27], was used in all mortar mixtures.

Table 3. Chemical and physical properties of lime

<table>
<thead>
<tr>
<th>Analysis</th>
<th>Value</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaO+Ca(OH)₂</td>
<td>85</td>
<td>%</td>
</tr>
<tr>
<td>CO₂</td>
<td>7</td>
<td>%</td>
</tr>
<tr>
<td>MgO</td>
<td>5</td>
<td>%</td>
</tr>
<tr>
<td>SO₃</td>
<td>2</td>
<td>%</td>
</tr>
<tr>
<td>Insoluble in acid+SiO₂</td>
<td>1.5</td>
<td>%</td>
</tr>
<tr>
<td>R₂O₃</td>
<td>0.5</td>
<td>%</td>
</tr>
<tr>
<td>Specific gravity</td>
<td>2.2</td>
<td>g/cm³</td>
</tr>
<tr>
<td>Bulk density</td>
<td>0.60</td>
<td>g/cm³</td>
</tr>
<tr>
<td>Sieve analysis, residue</td>
<td></td>
<td></td>
</tr>
<tr>
<td>200 µm</td>
<td>2</td>
<td>%</td>
</tr>
<tr>
<td>90 µm</td>
<td>7</td>
<td>%</td>
</tr>
<tr>
<td>Soundness</td>
<td>20</td>
<td>mm</td>
</tr>
<tr>
<td>Loss ignition</td>
<td>28.6</td>
<td>%</td>
</tr>
<tr>
<td>Penetration</td>
<td>10&lt;x&lt;50</td>
<td>mm</td>
</tr>
</tbody>
</table>
Three different types of clay were calcined separately at 550, 650, 750, and 850 °C in an electrically heated kiln (ÖZMAK with a capacity of max 1200 °C (±5)) without any other process and for each temperature, the clay samples were kept in the kiln for 30 minutes. After the samples cooled, they were ground and particle size analysis was carried out by sieving them with 250, 125, 90, 63, and 45 μm standard sieves. According to the ASTM C 618-03 Standard [26] at most 34% of the pozzolan would be retained when wet sieved on 45 μm. On the other hand, according to the ASTM C 593-95 Standard [28] a maximum of 2% of the pozzolan on 600 μm (No. 30) and a maximum of 30% of the pozzolan on 75 μm (No. 200) sieve is supposed to be retained. In the study, 78-90% of all the calcined clays used have smaller grain sizes than 45 μm, which also means a retention ratio of 10-22% on 45 μm. 5-15% of all the materials have grain sizes larger than 75 μm and smaller than 250 μm and fit well with the requirements indicated in either of the standards mentioned. Results of particle size analysis show that the granulometric curves and grain size distributions of all the samples display closely similar characteristics (Figure 1).

2.2. Samples produced and their curing conditions

Material amounts used in the mixtures were determined according to TS 25 Standard [29] (Table 4). Mortar mixtures were prepared in standard molds sized 40 x 40 x 160 mm.

Table 4. Proportions of the components of the mortar produced according to the TS 25 Standard.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Weight (in gram)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard sand</td>
<td>1350</td>
</tr>
<tr>
<td>Slaked lime Ca(OH)₂ (L)</td>
<td>150</td>
</tr>
<tr>
<td>Pozzolan (P)</td>
<td>2x150x (δP/δL)*</td>
</tr>
<tr>
<td>Water</td>
<td>0.50 (L+P)</td>
</tr>
</tbody>
</table>

*δP= specific density of pozzolan,  
δL= specific density of slaked lime

Curing conditions applied were in accordance with ASTM C 593-95 Standard. A total of 42 samples were prepared, with three samples for each mixture. 36 of these samples constitute the three series for three different types of clays (K1, K2, K3), calcined at four different temperatures. Thus, each of these clay series has four sets of three samples (Figure 2a). Two more mixtures, namely andesite rock powder added mortar (SM) and pure lime mortar (LM) were prepared and cured under similar conditions for comparison purposes, again with three samples being produced for each mixture.
After the samples were prepared, they were cured at 55 (±2) °C (RH= 90%) in a drying kiln for seven days as suggested by the ASTM C 593-95 Standard. Samples taken out of the drying kiln were cured at T= 23 (±2) °C, (RH= 60%) for four hours and then weighed on an electronic balance (PRESICA 4000 C) to determine their unit masses. Then, they were subjected to other tests. The results of the experiments are based on the assessment of the seven-day data.

2.3. UPV measurements, mechanical and physical experiments

Ultrasonic pulse velocity (UPV) measurements of all the mortars were determined according to the EN 14579 Standard [30]. Longitudinal wave measurements were taken by using PUNDIT (CNS Electronic Ltd.) non-destructive ultrasound equipment (transducer frequency with 54 kHz) (Figure 2b). The mechanical tests were done by the AMSLER Type 6DB7F120 hydraulic press with a capacity of 6-60 kN in 1.5 N/s. The prismatic specimens were applied to flexural strength tests first. Then those pieces surviving the flexural strength tests were applied to compressive and splitting tensile strength tests. Compressive strength, flexural strength and splitting tensile strength tests were performed following the standards of TS EN 1015-11 [31] and TS EN 12390 [32]. The EN 1939 [33] test method was used to determine the basic physical properties of specimens. Results of the tests are presented in Table 5.

Table 5. The properties of all the mortar series (for 7 days)

<table>
<thead>
<tr>
<th>Series</th>
<th>Firing temperature (°C)</th>
<th>Unit weight (g/cm²)</th>
<th>Open porosity (p) (%)</th>
<th>UPV (km/s)</th>
<th>E_d (kN/mm²)</th>
<th>Compressive strength (Rc) (N/mm²)</th>
<th>Flexural strength (Rf) (N/mm²)</th>
<th>Splitting strength (Rs) (N/mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>K1</td>
<td>550</td>
<td>1.98</td>
<td>29.6</td>
<td>1.97</td>
<td>2.76</td>
<td>1.21</td>
<td>1.54</td>
<td></td>
</tr>
<tr>
<td></td>
<td>650</td>
<td>1.98</td>
<td>29.5</td>
<td>2.10</td>
<td>8.92</td>
<td>3.17</td>
<td>1.31</td>
<td>1.58</td>
</tr>
<tr>
<td></td>
<td>750</td>
<td>2.03</td>
<td>26.5</td>
<td>2.94</td>
<td>17.86</td>
<td>6.89</td>
<td>2.96</td>
<td>3.37</td>
</tr>
<tr>
<td></td>
<td>850</td>
<td>2.06</td>
<td>26.8</td>
<td>2.92</td>
<td>17.85</td>
<td>6.27</td>
<td>2.73</td>
<td>3.06</td>
</tr>
<tr>
<td>K2</td>
<td>550</td>
<td>2.03</td>
<td>27.9</td>
<td>2.26</td>
<td>10.57</td>
<td>2.71</td>
<td>1.60</td>
<td>1.78</td>
</tr>
<tr>
<td></td>
<td>650</td>
<td>2.08</td>
<td>26.8</td>
<td>2.82</td>
<td>16.84</td>
<td>6.54</td>
<td>3.00</td>
<td>3.53</td>
</tr>
<tr>
<td></td>
<td>750</td>
<td>2.07</td>
<td>26.5</td>
<td>2.88</td>
<td>17.48</td>
<td>6.85</td>
<td>3.10</td>
<td>3.68</td>
</tr>
<tr>
<td></td>
<td>850</td>
<td>2.05</td>
<td>26.6</td>
<td>2.89</td>
<td>17.43</td>
<td>6.73</td>
<td>3.09</td>
<td>3.56</td>
</tr>
<tr>
<td>K3</td>
<td>550</td>
<td>1.99</td>
<td>28.4</td>
<td>2.23</td>
<td>10.06</td>
<td>3.12</td>
<td>1.50</td>
<td>1.73</td>
</tr>
<tr>
<td></td>
<td>650</td>
<td>2.05</td>
<td>28.3</td>
<td>2.44</td>
<td>12.46</td>
<td>4.13</td>
<td>1.70</td>
<td>2.08</td>
</tr>
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<td></td>
<td>750</td>
<td>2.05</td>
<td>27.5</td>
<td>2.59</td>
<td>14.04</td>
<td>4.38</td>
<td>1.90</td>
<td>2.28</td>
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<td></td>
<td>850</td>
<td>2.04</td>
<td>27.6</td>
<td>2.60</td>
<td>14.06</td>
<td>4.27</td>
<td>1.86</td>
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<td>SM</td>
<td>1.94</td>
<td>29.8</td>
<td>1.82</td>
<td>6.51</td>
<td>3.01</td>
<td>1.10</td>
<td>1.77</td>
<td></td>
</tr>
<tr>
<td>LM</td>
<td>1.74</td>
<td>31.2</td>
<td>1.11</td>
<td>2.17</td>
<td>1.10</td>
<td>0.48</td>
<td>0.75</td>
<td></td>
</tr>
</tbody>
</table>

Figure 2. (a) The mortars produced (b) Measurement of the ultrasound pulse velocity (UPV).
3. RESULTS AND DISCUSSION

3.1. Effect of calcined clays on pozzolanic activity and mechanical properties

Mechanical properties of all the mortar series produced are given in Figure 3. For pozzolanic activity, a minimum value of 4.0 N/mm$^2$ is specified for compressive strength by TS 25 Standard (this value is 4.1 N/mm$^2$ in ASTM C 593-95). Compressive strength of clay mixed samples calcined at 550°C is lower than this reference value. However, values for this parameter increase for the clay mixed samples calcined at temperatures greater than 550°C. As it can be seen from Figure 4, the highest compressive strength values were obtained by the clay mixtures calcined at 750°C. Mechanical behavior of clays calcined mixtures at 850°C are generally similar, though strength and pozzolanic activities are in a tendency to decrease.

![Figure 3. Mechanical properties of all the mortar series](image)

![Figure 4. Compressive strength (Rc) of clay mixed samples depending on the temperatures of calcinations](image)

The same relationship is available also for the flexural (Rf) and splitting tensile (Rs) strengths of the samples (Figure. 5). With the exception of lime mortar (LM), flexural strength (Rf) of all the samples are above the reference value of min 1.0 N/mm$^2$ specified in TS 25 standard. Maximum flexural and tensile splitting strength were obtained for samples calcined at 750°C. Flexural strength of the SM mixture, being 1.1 N/mm$^2$, is slightly above the limit value (Table 5).
Several studies published on pozzolanic properties of clays also state that pozzolanic activity decreases above 850-900 °C [17, 24] and this fact could be related to the re-crystallization of amorphous silica with increasing calcination temperature [34]. Clays calcined show mineralogical change with calcining temperature and under 800 °C generally acquire better pozzolanic properties [35, 36]. Therefore, the results of this study are compatible with the existing literature. In the case of rock powder added (SM) sample, prepared for comparison purposes and cured under similar conditions, values obtained for mechanical properties were low, because it did not contain minerals active enough to chemically react with Ca(OH)₂. Yet, the chemical composition of andesite powder contains oxide components in sufficient amounts for pozzolanic activity (SiO₂+Al₂O₃+Fe₂O₃=82%). Therefore, a high content of SiO₂ and other oxides in a pozzolan, while being a significant indicator of greater probability of presence of amorphous silica (SiO₂) in sufficient amount, may not definitely indicate that the substance has pozzolanic property. What is important for pozzolanic activity is a high ratio content of the amorphous SiO₂, rather than crystalline SiO₂.

In all these relationships, the bonding phase which gains a more rigid structure because of the calcium and alumina silicates that form as a result of the pozzolanic reaction taking place between Ca(OH)₂ and active clay minerals (especially in those clays calcined within 650-850 °C) certainly plays an important role. The strong chemical bonds formed by pozzolanic reaction on the interfaces of mortar components (aggregate/binder and binder/binder phases), increases the mechanical strength values of the mortar, by providing good cohesive power. This characteristic has been provided, despite their different pozzolanic properties, by the clay mixed samples of K1, K2 and K3 series calcined at 750 °C.

Mechanical properties of pure lime mortar (LM) have very low values as expected.

### 3.2. Effect of calcination on UPV and E₅

Depending on the calcination levels of clays, UPV values of the mortars prepared with these clays increase. UPV values of clay mixed samples vary between 1.97-2.94 km/s (Figure 6), and the E₅ values calculated by Eq.1 vary between 7.99-17.9 kN/mm² (Figure 7).
UPV values are 1.82 km/s in SM and 1.11 km/s in LM samples. UPV and $E_d$ values of all the mortars prepared by clays calcined at 550 °C are low, like their mechanical properties. Since the clay minerals calcined at this temperature had not gained sufficient pozzolanic characteristic, silicate formation and structural rigidity had not yet materialized in the internal structure of these mortars. The highest UPV and $E_d$ values were obtained for samples mixed with clays calcined at 750 and 850 °C. This temperature lies at the same time, within the interval suitable for pozzolanic activity. However no significant increase takes place in UPV values of mixtures containing clays calcined at 850 °C (Figure 8). This means that active minerals in clays calcined at high temperature are inclined to change phase and hence, their pozzolanic characteristics have begun to decrease. Consequently, chemical bonds between the mortar and matrix components become weakened, sound transmission time (t) of mortars get longer and ultrasonic speeds (V) end up having lower values.

As is seen, clays can have different pozzolanic activity depending on firing temperature and through UPV measurements valuable information can be obtained about the internal structure of mortars prepared with these clays. Therefore, it may be claimed that significant relations exist between pozzolanic activity of clays and the UPV and $E_d$ values of calcined clay mixed mortars. The characteristic of rigid internal structure acquired through pozzolanic reaction also shortens the transmission time of sound waves, thereby increasing the UPV values of these mortars. Experimental values indicate that silicate formation induced in the internal structure of pozzolan added mortars by pozzolanic reaction and pozzolanic activity can be traced with the UPV technique.

Similar relations are valid also for the SM series prepared by andesite powder of low pozzolanic activity and the UPV and $E_d$ values of the mortar produced by this material are lower than those of the high activity clay mixtures. And when the same relations are examined for lime mortars (LM) produced with hydrated lime, since no pozzolanic reaction takes place, lower UPV and $E_d$ values of these mortars are to be expected. The increase in the UPV values of lime mortars can vary in time depending on the carbonate formation level in lime. As known, carbonate formation
in lime proceeds at a slow rate depending on the CO₂ content of the environment. After long term investigations of UPV-carbonization relation in lime mortars, respective correlations can also be evaluated.

3.3. Relationships between UPV and mechanical properties of mortars

Relationships between the UPV and mechanical characteristics of mortars have been evaluated with respect to compressive, flexural and splitting tensile strengths (Rc, Rf, Rs) and significance of the correlations has been investigated. It is to be noted here that, the relationship between compressive strength (Rc) and UPV (V) for K1, K2, K3 series is more significant. Compressive strength being an indicator of pozzolanic activity in pozzolan added mortars, potential strong correlations would also indicate that the pozzolanic activity they acquire depending on the calcination temperature of clays can be determined with the UPV technique.

3.3.1. Correlation of UPV - compressive strength

UPV-Rc correlation has been investigated by using the Rc=f(V) relationship and applying linear regression to the data obtained from compressive tests of mortars. These evaluations have been carried out both for the calcined clay mixed mortars (K1, K2, K3) and also for all of the mortar series.

The linear regression coefficient (Rc) of the correlation between UPV and compressive strengths (pozzolanic activities) of the mortars produced with K1 and K2 clay samples have been determined as r²= 0.989 for K1; and r²= 0.996 for K2. For the mixtures of K3 clay sample this value is r²= 0.920 (Figure 9). High correlation coefficients indicate a strong relationship between compressive strengths and UPV values of these calcined clay mixed mortars.

When the UPV-Rc correlation is evaluated for calcined clay added mixtures (K1, K2, K3) and for all the series together, the linear regression coefficient takes on the relatively low values of R² K1,K2,K3, r²= 0.908 and R² All series, r²= 0.854 (Figure 10).
The small deviation in the linear regression coefficient for $R_c$ in $K_{1,2,3}$ series can be considered normal both due to the mortar samples having anisotropic structure and the different clay samples used in their mixtures display different pozzolanic behaviors according to their temperature intervals of calcination, depending on their mineralogical structure. Because of their high correlation coefficients, calcined clay mortar series independently within themselves, indicate the strong correlation in between UPV-$R_c$.

### 3.3.2. Correlations of UPV- flexural and UPV- splitting tensile strength

Likewise, relationships between UPV- flexural strength ($R_f$) and UPV- splitting tensile ($R_s$) strength have been investigated using experimental data with linear regression, and the evaluations are made under conditions similar to those of the compressive strength tests.

Here too, a strong relationship can be established between UPV and flexural strengths ($R_f$) of mortars. As with the case of compressive strengths, the correlation between UPV and flexural strength has high values for every clay mixed mortar group within itself ($K_1$, $r^2 = 0.989$; $K_2$, $r^2 = 0.998$; $K_3$, $r^2 = 0.982$) (Figure 11). Correlation coefficient between UPV and $R_f$ for all of the calcined clay mixed samples ($R_f$ for $K_{1,2,3}$) is  $r^2 = 0.894$ and when the same relationship is evaluated collectively for all of the mortar series, the correlation coefficient ($R_f$ for All series) turns out to have the relatively lower value of $r^2 = 0.872$ (Figure 12).

![Figure 11](image1.png)  
Figure 11. The correlation between UPV and flexure strength of each mortar group including calcined clays mixture

![Figure 12](image2.png)  
Figure 12. The correlation between UPV and flexure strength of all the mortar series pozzolan mixed and unmixed.

Relationship between UPV and splitting tensile strength ($R_s$) has been evaluated, according to the same $R_s=f(V)$ expression, by linear regression based on average values for calcined clay mixed and all the mortar series. Here too, coefficients of regression between UPV and $R_s$ for calcined clay mixed mortar series within themselves have high values as ($R_s$ for $K_1$ $r^2 = 0.979$; $R_s$ for $K_2$ $r^2 = 0.992$; $R_s$ for $K_3$ $r^2 = 0.967$) (Figure. 13) and indicate a strong correlation. The correlation coefficients take on slightly lower values of ($R_s$ for $K_{1,2,3}$) $r^2 = 0.877$ when the UPV and splitting tensile strengths of clay mixed mortars are evaluated together and ($R_s$ for All series) $r^2 = 0.829$ when the same evaluation is made for all mortar series together (Figure 14).
As is seen, the UPV- flexural (Rf) and UPV- splitting tensile (Rt) strength correlations are close to the UPV-compressive (Rc) strength correlation and this indicates the presence of a relatively similar behavior. Therefore, with respect to all mortar series, the correlations between UPV and (Rc), (Rf) and (Rt) can be established with certain reliability. However, this correlation is relatively weaker and this fact might be stemming from the samples not having a homogenous internal structure and the LM mixture, for which pozzolanic reaction is of no issue, not being a representative of the relationship between the relevant characteristics. It would be more appropriate to evaluate the relationships between UPV and the three strength characteristics after measurements with more samples and longer periods of curing.

Nevertheless, due to high correlation coefficients, the relationship between UPV and the mechanical strengths (Rc, Rt, Rf) is more strongly established within each calcined clay mixed K1, K2, K3 series. The strong correlation found between UPV and these three strength characteristics for mortars produced with clays calcined at different temperatures, thus having different pozzolanic characteristics, indicates that these correlations can be used as a reliable measurement tool in determination of pozzolanic activity level by UPV. As a result of these evaluations, possibility of monitoring pozzolanic activity with non destructive UPV technique can be safely suggested.

### 3.3.3. Correlations of UPV-porosity and porosity-mechanical characteristics

For all the mortars, apparent porosity ratios (p) determined from water absorption values vary between 26.5 % and 32.3 %. The water/binder ratio is the most important parameter influencing porosity. Pulse velocity decreases with increasing porosity, and it increases with water content [37, 38]. In a previous study, accurate estimates of porosity were made from measurements of ultrasonic pulse velocity [39, 40]. However, porosity of lime-pozzolan mortars decreases with time and is strongly influenced by the curing conditions [41]. Therefore, all mixtures produced for this study were cured under similar conditions.

Mortars with higher porosity give lower UPV values. There is an inverse relationship between UPV and porosity for all of the mortar series. Existence of an inverse relationship between porosity and UPV is stated also in the literature [3, 37, 40].
When the relationship between porosity (p) and UPV (V) values is investigated according to \( V = f(p) \) formulation, the linear regression coefficient was found to be \( r^2 = 0.933 \) for all of the calcined clay mixed mortars and \( r^2 = 0.955 \) for all mortars together (Figure 15). The data indicates the presence of a generally strong correlation between the UPV and porosity values of mortars.

\[
\begin{align*}
\text{Lin.Regr. For All Samples} & : y = -2.7342x + 34.638 \quad R^2 = 0.955 \\
\text{Lin.Regr. For K1,K2,K3} & : y = -3.0958x + 35.584 \quad R^2 = 0.933
\end{align*}
\]

Figure 15. The correlation between porosity and UPV all mortar series of calcined clay mixed, and unmixed

Correlation coefficient between porosity (p) and compressive strength (Rc) is found to be \( r^2 = 0.916 \) for all the mortar series together, and as \( r^2 = 0.889 \) for clay mixed (K1, K2, K3) series (Figure 16). Papayianni and Stefanidou [41], had investigated by a model the relationship between porosity and compressive strength in lime-pozzolan mortars and determined the regression coefficient between porosity and strength as \( r^2 = 0.884 \). Regression coefficient for the K1, K2 and K3 series being close to this value indicates agreement with that study and the existence of a similar relationship.

\[
\begin{align*}
\text{Lin.Regr. For K1,K2,K3} & : y = -0.6622x + 30.98 \quad R^2 = 0.889 \\
\text{Lin.Regr. For All Samples} & : y = -0.7653x + 31.576 \quad R^2 = 0.916
\end{align*}
\]

Figure 16. The correlation between porosity and compressive strength of all mortars.

When the same evaluation is done in relation to porosity and other mechanical characteristics (Rf, Rs) of all mortars, linear regression coefficients between porosity (p) and flexural (Rf) and splitting tensile (Rs) strengths are found to be \( R^2_{\text{All series}} = 0.903 \) and \( R^2_{\text{All series}} = 0.849 \) (Figure 17). These values may be considered to be close to the correlation coefficient between (p) and (Rs). These results are also indicated the presence of a relatively good correlation also between porosity and mechanical characteristics of mortars. The strength of lime-pozzolan mortars depends on the pozzolanicity index of the pozzolan which is a measure of the lime-pozzolan reactivity. Furthermore, the structure of the porosity system is another factor effecting the strength-porosity relation [41].
4. CONCLUSIONS

Results of this experimental study can be summarized as follows:

1- There is generally a close relationship between UPV values and mechanical ($R_c$, $R_f$, $R_s$) properties of calcined clay added mortars. Mechanical properties of lime mortars with pozzolan and pozzolanic characteristic of the admixture can be reliably determined by means of the UPV test method.

2- Reliable information about pozzolanic property can be obtained by measuring the UPV of lime mortars produced with pozzolanic materials. Although the coefficients in the empirical expressions may change according to the pozzolan type, general trend remains almost the same.

3- There is a high degree of correlation between the UPV values and pozzolanic properties of lime mortars produced with calcined clays.

4. There is a good correlation between the pozzolanic activities calcined clays acquire according to the range of temperatures at which they are calcined and the UPV values of lime mortars produced with them.

5- Even when a substance has low pozzolanic properties, this affects the UPV of the mortars produced with this substance and in turn may give information about the pozzolanic properties of the substance. The UPV values of the mortars produced with low or no-pozzolanic materials are low.

6- Generally, a good correlation exists between porosity ratio and UPV values of lime mortars and pozzolan added lime mortars. This holds also for porosity ratios of these mortars and their mechanical characteristics.

To summarize, pozzolanic activities of calcined clays can be determined with measurements of UPV of the mortars prepared with these materials. As further complementary work, application of the same method to cement binder composites produced with natural or other artificial pozzolans may be suggested.

REFERENCES


