Effect of Carbonation on the Collapse Potential of Magnesium Oxide Treated Gypseous Soil

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1. INTRODUCTION

Gypseous soil is one of the most problematic soils which is subjected to collapse under both of saturation and loading. It covers about 27 - 36% of Iraq area [1].

Collapsible soils are characterized by very distinct geotechnical properties that include high void ratio, low initial bulk density and water content, great dry strength and stiffness, high percentage of fine grained particles and zero or slight plasticity. The main geotechnical problem associated with these soils is the significant loss of shear strength and volume reduction occurring when they are subjected to additional water from rainfall, irrigation, broken water or sewer lines, moisture increase due to capillarity or ‘’pumping’’ as a result of loading, ground water rise, etc. Generally, collapsible soils are under unsaturated conditions in the dry state, with negative pore pressure resulting in higher effective stresses and greater shear strength. Cases of wetting induced collapse in gypseous type soils have been documented in natural deposits and in man-made fills. In the latter case they can often cause large differential settlements that reduce the serviceability of the structure, and raise the frequency and cost of rehabilitation.

Many researches dealt with improving the collapsible gypseous soil with different materials to reduce the collapsibility of the soil such as lime, cement, silica fume etc. Currently, various types of recyclable materials are used in civil engineering applications. One of the future challenges in civil engineering field facing sustainability and the bulk utilization of waste materials without affecting the performance of the product related to civil engineering field [2].

The work conducted by Hayal et al. [3] was using nano silica and nano clay with different percentages to improve the gypseous soil brought from Bahr Al-Najaf.

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**Table 1.** Summary of research

| Research/Project | Methodology | Result/Findings |
|------------------|-------------|-----------------|
| Hayal et al. [3] | Nano silica and nano clay | Improved the gypseous soil brought from Bahr Al-Najaf |

**Table 2.** Comparison of different materials

| Material Type | Improvement |
|---------------|-------------|
| Lime | Moderate improvement |
| Cement | Significant improvement |
| Silica fume | Very significant improvement |

**Figure 1.** Comparison of different materials improvement

*Image*
The results showed that using nano clay reduced the collapse potential about 73.75%.

Abdulrahman et al. [4] used different percentages of fiber plastic to improve the collapsibility of the gypseous soil and they have concluded that 1% of fiber plastic reduce the collapse potential from 12.5% to 0.96%.

Large scale reinforcement of the collapsible soil by using encased stone column was performed by Bahrami and Marandi [5] with aspect ratio (diameter to length) 10 to 25%. They have concluded that the optimum aspect ratio is about 15% which reduces the collapse potential about 82%.

One of the recent developments in additive materials is the reactive magnesia or reactive magnesium oxide (MgO). Magnesium oxide is a white solid material that occurs naturally as periclase, it is a source of Magnesium. The magnesium oxide is not virulent but it is one of the materials friendly with environment [6, 7].

Some researches used magnesium oxide and carbonated magnesium oxide for stabilizing soil and compared results with lime, cement etc. Carbonated magnesium oxide is adequate with sand and porous blocks, there were laboratory studies by treating sandy soil with 5 and 10% magnesium oxide; then, the mixture is subjected to carbon dioxide gas for about 3 hours to accelerate the treating and the results were compared with Portland cement. When the carbon dioxide increased, the pressure that caused increasing in carbonation till reaching improvement percent about 20% then increasing the carbonation did not reveal a clear increasing in strength. The strength will be at the same range when the carbonation period was about 2 days [8].

Yi et al. [9] studied the treatment of silty soil with two percentages of magnesium oxide and carbonated at different period times 0.5 hour, 6 hours and 7 days with three pressures of carbonation 50, 100 and 200 kPa. The results showed that the pressure of carbonation increased the strength of the soil but no clear increasing in strength with increasing pressure. The work confirmed that the carbon dioxide is rapiding the magnesium oxide treatment, as well as this method is adequate to soil improvement (stabilization and solidation).

Liu et al. [10] used the unconfined compressive strength to investigate the improvement of the clayey silt soil with different percentages of 12 hours carbonation magnesium oxide. The results showed that the unconfined compressive strength increased and the structure of the soil after treatment changed from elastic-plastic to brittleness.

Hwang et al. [11] studied the improvement of the silty sand sediment in South Korea by using unconfined compressive strength of soil treated with 30% of carbonated magnesium oxide. The results illustrated that the strength after 1 year was 4.78 MPa which is higher than Portland cement about 1.3 times.

Cai et al. [12] depended on cycles of drying and wetting to mixing of silty soil with 15 % of carbonated Magnesium Oxide for curing periods 3 and 6 hours then compared this 15% Portland cement. The results showed that the strength of carbonated magnesium oxide higher than on cement as well as at carbonated magnesium oxide, the durability of the soil increased with the cycles of drying and wetting and curing period for 3 hours gave results similar to 6 hours.

Wang et al. [13] depended on mixing a clayey soil with different percentages of magnesium oxide, calcium oxide and fly-ash then carbonated the mixture. The results showed that the strength after carbonating period time 0.5 hour was about 75% as compared with the strength in the soil stabilized with 10% Portland cement after 28 days. The optimum time of carbonation was 6 hours.

The main purpose of this study is to investigate the effectiveness of curing period time of carbonation on magnesium oxide stabilization of gypseous soil by using different percentages of magnesium oxide prepared at two relative densities. This study was done by conducting conventional tests (single oedometer test and double oedometer test) and modified collapse test.

2. MATERIALS

2.1. Soil The granular soil utilized in this study was supplied from Tikrit, north of Baghdad, and had a gypsum level of 49%. Table 1 shows the physical parameters, whereas Figure 1 shows the grain size distribution. The specifications of the American Society for Testing and Materials were adopted. The Unified Soil Classification System classifies the soil as poorly graded sand (SP).

2.2. Magnesium Oxide The magnesium oxide used in this research has a light weight with a unit weight of 1 kN/m³. Magnesium oxide, with the chemical formula MgO, is a common alkaline earth metal oxide. White powder with a melting point of 2852°C and a boiling temperature of 3600°C, as well as a relative density of 3.58 (at 25°C). It can be dissolved in acid or ammonium salt solutions. It produces magnesium hydroxide when it reacts slowly with water, having a solubility of 0.01 g/L. It can be made into magnesium bicarbonate by dissolving it in a carbon dioxide aqueous solution.

2.3. Carbonation The carbonation is the carbon dioxide pressure made by using carbonation apparatus. The apparatus was manufactured form several parts as illustrated in Figure 2. The main purpose of carbonation is to stabilize the improvement of magnesium oxide in the soil.
TABLE 1. Physical properties of soil

| Physical properties | Value |
|---------------------|-------|
| Gypsum content (%)  | 49    |
| Specific gravity (Gs)| 2.41  |
| Liquid limit (L.L) (%) | 26    |
| Plastic limit (P.L) (%) | N.P   |
| Gravel (%)          | 0     |
| Sand (%)            | 96    |
| Fines (%)           | 4     |
| D_{60} (mm)         | 0.4   |
| D_{10} (mm)         | 0.2   |
| D_{30} (mm)         | 0.11  |
| Uniformity coefficient (Cu) | 3.64 |
| Curvature coefficient (Cc) | 0.91 |
| (O.M.C) (%)         | 12    |
| $\gamma_{\text{dry max.}}$ (kN/m$^3$) | 17.45 |
| $\gamma_{\text{dry min.}}$ (kN/m$^3$) | 12.12 |
| Classification      | SP    |

The carbonation curing apparatus was used to apply a low pressure of pure carbon dioxide gas on soil samples. The major components of the set-up include compressed gas tanks, pressure vessel, thermocouple, data acquisition, vacuum and pressure transducer. A gas injection-releasing carbonation process was used for all carbonation tests.

The soil carbonation procedure is made as follows:
1. Mixing the soil with using a percentage of magnesium oxide carefully.
2. Placing the mix in the carbonation container if the test needs a carbonation process.
3. The valve of vacuum is turned on and the electric power of vacuum is on to extract the air from the soil and container.
4. The valve of vacuum is closed and opens the valve of carbon dioxide till reaching the same pervious pressure.

5. The carbon dioxide valve is turned off and the curing continues for a period of time.

The carbonation curing apparatus was used to subject samples to a low pressure of pure carbon dioxide gas and oxygen gas. A gas injection-releasing carbonation process was used for all carbonation tests [14].

3. CHEMICAL REACTIONS

The carbonation of any structure is provided enough of carbon dioxide to penetrate through its porous, reactive magnesium oxide hydrates by this equation as described by Al-Tabaa [8] and Yi et al. [9]:

\[
\begin{align*}
\text{MgO} + \text{H}_2\text{O} & \rightarrow \text{Mg(OH)}_2 \text{ (brucite)} \\
\text{Mg(OH)}_2 + \text{CO}_2 + 2\text{H}_2\text{O} & \rightarrow \text{MgCO}_3 \cdot \text{H}_2\text{O} \text{ (nepside}) \\
5\text{Mg(OH)}_2 + 4\text{CO}_2 + 2\text{H}_2\text{O} & \rightarrow (\text{Mg})_4(\text{CO}_3)_2(\text{OH})_6 \text{ (5Hype)} \\
5\text{Mg(OH)}_2 + 4\text{CO}_2 & \rightarrow (\text{Mg})_4(\text{CO}_3)_2(\text{OH})_6 \cdot 4\text{H}_2\text{O} \text{ (hydromagnesite)}
\end{align*}
\]

4. TESTING PROGRAM

The testing program of the gypseous soil treated with magnesium oxide and carbonated magnesium oxide is presented in Figure 3.

4. 1. Single Oedometer Test (SOT) This type of test depends on loaded the sample at initial water content till reached the vertical stress to 200 kPa then the sample soaked with water for 24 hours. The difference between
the soaked and unsoaked settlement results represents the collapse potential. After that, the test is continued the same as consolidation test from loading and unloading.

4.2. Double Oedometer Test DOT 

This test can be performed with two identical samples: the first is tested at its natural water content until the end of the test, while the second sample is soaked at the start. The testing procedure is the same as the consolidation test procedure. At any given pressure, the difference between the two curves of the void ratio - pressure (e - log P') denotes soil collapse. Collapse potential is evaluated using a typical oedometer instrument in a constant temperature and humidity condition, according to ASTM D5333 [15].

4.3. Modified Collapse Test MCT

There are some weaknesses in the traditional methods (single oedometer test and double oedometer test) such as the size of sample is too small to measure the collapse and saturation method from up by downward flow that may not making saturation for all the soil particles and do not show the right collapse potential; therefore, a new type of tests that is proposed in this study and used to calculate the collapse potential and compare the results with the single and double oedometer results.

The dimensions of a modified Rowe cell are employed in the test. An electric motor with a load cell and indication is used to load a load with a diameter of 150 mm and a height of 50 mm. The sample is loaded in the same way as a single oedometer test until it reaches a vertical stress of (200 kPa), after which it is soaked with water from the bottom up using a scaling tank filled with water with a hydraulic gradient of 3.667 by a hydraulic height of 170 mm above the soil sample and monitoring the saturation point with SMCS sensors [16].

The frame of loading consists of a motor with piston to shed a required load with a gear box to control the rate of velocity. Load cell of 5 ton capacity is used with its indicator to record the load with a dial gauge to record the settlement. The load is applied gradually over a short period of time every 24 hours, and the settlement is recorded every 15 minutes with the time the loading is calibrated.

The test employs two filter sheets to prevent gypsum salt that has not dissolved in water from entering the porous stone holes. The sample is soaked for 24 hours after saturation to document any additional settlement. The test then continues with more loading and unloading, as in a traditional consolidation test. Figure 4 illustrates the all the parts of modified collapse test (MCT).

5. RESULTS AND DISCUSSION

The effect of carbonation time on the collapse potential for the conventional tests (SOT and DOT) for different percentages of magnesium oxide and relative densities are described in Figures 5 to 8. A summary of all collapse test results is illustrates in Table 2.

The effect of carbonation time on collapse potential for the modified collapse test (MCT) for different
Figure 5. Change in collapse potential with increase of carbonation period time for 0% magnesium oxide

Figure 6. Change in collapse potential with increase of carbonation period time for 5% magnesium oxide

Figure 7. Change in collapse potential with increase of carbonation period time for 10% magnesium oxide

Figure 8. Change in collapse potential with increase of carbonation period time for 15% magnesium oxide

TABLE 2. Summary of the collapse potential of all tests

| Collapse Potential | CO₂=0 hr | CO₂=1 hr | CO₂=3 hrs | CO₂=24 hrs |
|--------------------|---------|---------|-----------|-----------|
|                    | MgO (%) | MCT (%) | SOT (%)  | DOT (%)  | MgO (%) | MCT (%) | SOT (%) | DOT (%)  | MgO (%) | MCT (%) | SOT (%) | DOT (%)  | MgO (%) | MCT (%) | SOT (%) | DOT (%)  |
| R.D 35 (%)         |         |         |          |          |         |         |          |          |         |         |          |          |         |         |          |          |
| 0                  | 22.3    | 15.8    | 12.25    | 0        | 11.11   | 7.1     | 4.65     | 0        | 10.28   | 5.58    | 4.05     | 0        | 9.97    | 5.08    | 3.95     |
| 5                  | 9.07    | 6.6     | 3.1      | 5        | 6.17    | 4.6     | 3        | 5        | 3.88    | 3.02    | 2        | 5        | 3.75    | 2.95    | 2        |
| 10                 | 4.78    | 3.8     | 0.3      | 10       | 3.85    | 2.25    | 0.005    | 10       | 2.32    | 1.97    | -0.85    | 10       | 2.24    | 1.96    | -1.55    |
| 15                 | 2.39    | 1.33    | -0.85    | 15       | 2.36    | 1.52    | 0.56     | 15       | 1.78    | 1       | -0.4     | 15       | 1.67    | 1       | -0.5     |

| R.D 75 (%)         |         |         |          |          |         |         |          |          |         |         |          |          |         |         |          |          |
| 0                  | 19.93   | 14.8    | 10.75    | 0        | 8.95    | 4.05    | 1.85     | 0        | 5.8     | 3.38    | 1.67     | 0        | 5.22    | 3.1     | 1.67     |
| 5                  | 3.02    | 1.75    | 1.35     | 5        | 2.65    | 1.73    | 1.31     | 5        | 1.99    | 1.6     | 0.925    | 5        | 1.73    | 1.46    | 0.925    |
| 10                 | 2.67    | 1.6     | -0.45    | 10       | 2.12    | 1.35    | -0.4     | 10       | 1.5     | 1.09    | -2.6     | 10       | 1.22    | 1.02    | -1       |
| 15                 | 1.92    | 0.75    | -0.35    | 15       | 1.93    | 1.2     | -1.6     | 15       | 1       | 0.75    | -1.73    | 15       | 0.96    | 0.6     | -1.5     |
percentages of magnesium oxide is described in Figures 9 and 10 for soils prepared at relative density 35 and 75%, respectively. A summary of all collapse test results illustrates in Table 2. This result agrees with the finding of Cai et al. [12], who found that magnesium oxide with 3 hours carbonated period time in silty soil was better than 6 hours.

Figures 11 and 12 illustrate the volume of water that is needed in the sample till 100% saturation with increasing the period time of carbonation for relative density 35% and 75%, respectively. Figures 13 and 14 illustrate the time of saturation with an increasing the period time of carbonation for relative density 35% and 75%, respectively. A summary of time and volume of water that needed to saturate are illustrated in Table 3.

When the soil is soaked and subjected to constant stress $\sigma_v$, large volume changes and sudden collapses occur. Soil settlement happened when the results of the dissolving of cementing bonds of gypsum, that is resulting the significant increasing in the soil compressibility. According to the movement of the
Papadopoulos et al. [17] reported on his soaked samples to check their dissolution that in the first hour of saturation early increase of mass was obtained and then with logarithm of time almost regular solubility rate can be noticed.

The main points of collapse potential in this research can be summarized as follows:

All test results showed increasing in collapse

1. Potential as compared with conventional tests for both of single oedometer test and double oedometer test.
2. Carbonation of collapsible gypseous soil without using magnesium oxide for the natural soil showed a noticeable decrease in collapsibility in the order of 200-250%.
3. For the carbonation period time, for all percentage of magnesium oxide, when increasing the carbonation time from 0 hour to 3 hours, there is a good reduction in collapse potential (about 1.5 to 2%), but there is no noticeable reduction in collapse potential when increasing the carbonation time from 3 hours to 24 hours.
4. At relative density 35%, the collapse potential for magnesium oxide 10% and 15% gave nearest value at the same time of carbonation period. At relative density 75%, the collapse potential for all magnesium oxide percentages gave nearest value at the same time of carbonation period.
5. The time and the volume of water that are needed for saturation were increasing with increasing both of magnesium oxide percentages and time of carbonation periods till reached carbonation 3 hours then became about steady line.
6. For conventional tests, the results of the collapse potential decreased when adding magnesium oxide which agreed with Shakir’s [18] reported data when adding cutback asphalt to gypseous soil the using of magnesium oxide in this study decreased the collapse potential about 5 times as compared with cutback asphalt reported by Al-Hadidi and Al-Maamori [19] using cement to gypseous soil the using of magnesium oxide in this study decreased the collapse potential about 1.5 times as compared with cement.

Because the density of the soil reduces with an increase in volume, the time required for saturation increases faster than the time required for saturation of the same model under the initial load [20, 21].

6. CONCLUSIONS

From the results of collapse tests carried out on gypseous soil treated with magniseoum oxide and subjected to different periods of carbonation, it can be concluded that:

- The carbonation of the soil treated with magnesium oxide accelerated the improvement of the gypseous soil and decreased collapse.
- Carbonation of collapsible gypseous soil without using magnesium oxide for the natural soil showed a

### Table 3. Summary of time and volume of water needed to saturate the samples

| CO₂=0 hr | CO₂=1 hr | CO₂=3 hrs | CO₂=24 hrs |
|---------|----------|-----------|------------|
| MgO (%) | Time for saturation (min.) | Volume of water (cm³) | MgO (%) | Time for saturation (min.) | Volume of water (cm³) | MgO (%) | Time for saturation (min.) | Volume of water (cm³) |
|---------|----------|-----------|------------|
| 0       | 15       | 250       | 0          | 21       | 259.4      | 0          | 23       | 263.9      | 0          | 23       | 365.9      |
| 5       | 41       | 386.5     | 5          | 45       | 397.3      | 5          | 50       | 412.7      | 5          | 52       | 415.1      |
| 10      | 55       | 450.8     | 10         | 62       | 471.7      | 10         | 66       | 487.1      | 10         | 69       | 489.4      |
| 15      | 86       | 492.3     | 15         | 90       | 511.2      | 15         | 97       | 528.6      | 15         | 100      | 531.1      |

| CO₂=0 hr | CO₂=1 hr | CO₂=3 hrs | CO₂=24 hrs |
|---------|----------|-----------|------------|
| MgO (%) | Time for saturation (min.) | Volume of water (cm³) | MgO (%) | Time for saturation (min.) | Volume of water (cm³) | MgO (%) | Time for saturation (min.) | Volume of water (cm³) |
|---------|----------|-----------|------------|
| 0       | 18       | 357.1     | 0          | 25       | 362.8      | 0          | 26       | 368.1      | 0          | 27       | 369.9      |
| 5       | 60       | 447.7     | 5          | 63       | 460.3      | 5          | 67       | 474.6      | 5          | 68       | 478.1      |
| 10      | 83       | 500       | 10         | 94       | 521.7      | 10         | 97       | 535.2      | 10         | 98       | 539.5      |
| 15      | 109      | 535.7     | 15         | 116      | 555.5      | 15         | 120      | 562.6      | 15         | 120      | 566.8      |
noticeable decrease in collapsibility in the order of 200 – 250%. The carbonation on untreated gypseous soil decreased the collapse potential more than 50% for modified collapse test and for the two relative densities, and more than 55% for conventional tests.

- The carbonation periods 3 and 24 hours revealed closely equal results for all tests; therefore, the optimum carbonation period is 3 hours.
- Using the modified collapse test, the time needed to saturate the sample and volume of water required for sample saturation increased with increasing the carbonation period time. The increase is about 50% and 5% for time and volume of water, respectively for untreated soil.
- When the soil is treated with 10% magnesium oxide and carbonated for 3 hours, the increase in time and volume of water is around 20% and 4%, respectively.
- Carbonation of the collapsible gypseous soil treated with magnesium oxide for a period of 3 hours resulted in a good reduction in collapse potential (about 1.5 to 2%).

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چکیده
پستانسیل ریزش از زمان دوره کربناته استفاده می‌شود و نتایج نشان داد که برای دوره بیش از 3 ساعت هیچ تغییر واضحی در پستانسیل ریزش وجود ندارد.