Study on the optimum process of microwave technology for soil treatment based on orthogonal experiment

Dengsheng Ma, Shuai Zhang, Qi Qing and Xirui Lu*
Key Subject Laboratory of National Defense for Radioactive Waste and Environmental Security, Southwest University of Science and Technology, Mianyang 621010, P. R. China
*Corresponding author: luxiruil16@163.com

Abstract. In order to improve the efficiency of microwave technology on soil solidification, three factors of sintering temperature, holding time and particle size were selected to test the conditions, and the solidified body were systematically studied by XRD and SEM. The results showed that the optimal conditions were of the sintering temperature is 1300 ℃ and the holding time is 30 min. Moreover, there was no significant effect on the solidification of the soil in the selected particle size range of 60-200 mesh. The studies showed that the optimal conditions technological process of soil solidification is 1300 ℃ for 30 min, particle size of 60-200 mesh, which could guide future researchers on dispose of contaminated soil solidification by microwave technology.

1. Introduction
Soil is the foundation of human survival. The soil pollution is becoming more and more serious in different ways due to the development of industry [1-3]. Several technologies are currently available for dispose of contaminated soil on the large scale [4-6]. However, many of the technologies lack flexibility in disposal contaminated soil, and suffer from high capital and operating costs. In this work, an alternative method that uses microwave technology to disposal the contaminated soil was studied. Microwave sintering provides a novel, fast, clean, and energy-efficient method for preparation of many inorganic solids [7]. More specifically, the microwave heating is generated by means of internal polarization molecular vibration, instead of an external heating source. Hence, the energy is directly transferred to the material through the interaction of electromagnetic waves with molecules leading to heating [8]. Moreover, increasing evidence shows that the microwave sintering technology is rapid and efficient [9]. Therefore, microwave sintering technology is being widely utilized to various fields in recent times [10-12].

In general, the influence of sintering temperature was very important on the optimum process of microwave technology for soil treatment. In order to further improve the efficiency of microwave technology on soil solidification, the orthogonal experiment method was applied to this work, and three factors of sintering temperature, holding time and particle size were selected to test the conditions.

2. Experimental method

2.1 Material preparation and characterization
In this paper, the natural soil (Xinjiang Malan peripheral nuclear test site) are chosen as substrate of
raw materials, the particle size of 60 mesh, 80 mesh, 100 mesh, 150 mesh and 200 mesh were selected by screen, and the soil samples were dried at 150 °C keep 12 h. Firstly, 10 g samples were pretreated with different temperatures for determine the appropriate temperature range and the sintering results are shown in Figure 1. According to the orthogonal test design, weigh up the samples of equal quality use analytical balance, and placed in the same type of corundum crucible and compacted, high temperature sintering in a microwave oven.

The phase structures of the samples were characterized by X-ray diffractometer (X’Pert PRO, Netherlands) with Cu Ka, Pipe pressure 40 Kv. The micro-structure of the sintered samples was observed by scanning electron microscope (FESEM, Ultra 55, Germany).

2.2 Orthogonal experiment
Sintering temperature, holding time and particle size are of great influence in microwave curing process of soil from single-factor research made. Meanwhile, to ensure test comprehensive, choose three factors and five levels (in table 1), that is \( L_{25}(5^3) \) to optimize the test.

| Table 1. Orthogonal experimental factors level Settings. |
| --- |
| Level | Factor | Temperature (°C) | Time (min) | Particle Size (mesh) |
| 1 |  | 1100 | 15 | 60 |
| 2 |  | 1150 | 30 | 80 |
| 3 |  | 1200 | 60 | 100 |
| 4 |  | 1250 | 120 | 150 |
| 5 |  | 1300 | 240 | 200 |

3. Results and discussion

3.1 Pretreatment analysis
Figure 1 shows the photos of soil before and after sintering. As we can see from the photos that the soil was almost unchanged at 600 °C, and the hardening phenomenon occurred when the temperature rises to 900 °C - 1000 °C, and the vitrification phenomenon occurred when the temperature rises to 1100 °C, and the vitrification phenomenon is more obvious at 1200 °C. So, the temperature range 1100 °C ~ 1300 °C were chosen as the sintering temperature range of orthogonal experiment, the temperature gradient is 50 °C as shown in table 2.

![Figure 1. Photos of soil before and after sintering](image)

| Table 2. Results of experiment \( L_{25}(5^3) \). |
| --- |
| Factor | Temperature (°C) | Time (min) | Particle size (mesh) |
| Test 1 | 1100 | 15 | 60 |
| Test 2 | 1100 | 30 | 80 |
| Test 3 | 1100 | 60 | 100 |
| Test 4 | 1100 | 120 | 150 |
| Test 5 | 1100 | 240 | 200 |
| Test 6 | 1150 | 15 | 80 |
| Test 7 | 1150 | 30 | 100 |
| Test 8 | 1150 | 60 | 150 |
Test 9 & 1150 & 120 & 200 \\
Test 10 & 1150 & 240 & 60 \\
Test 11 & 1200 & 15 & 100 \\
Test 12 & 1200 & 30 & 150 \\
Test 13 & 1200 & 60 & 200 \\
Test 14 & 1200 & 120 & 60 \\
Test 15 & 1200 & 240 & 80 \\
Test 16 & 1250 & 15 & 150 \\
Test 17 & 1250 & 30 & 200 \\
Test 18 & 1250 & 60 & 60 \\
Test 19 & 1250 & 120 & 80 \\
Test 20 & 1250 & 240 & 100 \\
Test 21 & 1300 & 15 & 200 \\
Test 22 & 1300 & 30 & 60 \\
Test 23 & 1300 & 60 & 80 \\
Test 24 & 1300 & 120 & 100 \\
Test 25 & 1300 & 240 & 150 \\

3.2 XRD analysis

Figure 2 shows the XRD patterns of sintered samples under different conditions. And all patterns are of typical XRD amorphous, except some crystal phases exist in samples. As shown in Figure 2 (a), the crystal phases has a large proportion, which is mainly composed of SiO$_2$ and NaAlSi$_3$O$_8$. However, the proportion of crystal phases gradually decreased to disappear with increasing sintering temperature as shown in Figure 2 (e). In addition, the phase structure was not obvious changed at the same temperature holding over 30 min. Therefore, temperature plays an important role in the vitrification process of soil than time. When the temperature is constant, the crystallization rate does not change with the increase of holding time. The results show that the highest amorphization rate is due to the increased temperature, and the best temperature is 1300 ℃, which the crystal phases has totally disappeared at holding 30 min.
3.3 Morphology analysis

Figure 3 shows the typical SEM image of solidified samples by microwave at 1100 °C-1300 °C for 30 min. As can be seen that the circular holes were found in the soil solidified body surface at 1100 °C, 1150 °C, 1200 °C, and with the increase of temperature, the circular holes distribution rate decreases in the equal-area zones and it completely disappears as shown that in Figure 3 (d), (e). The change of the circular holes in the solidified soil can be regarded as a phase transfer phenomenon, the bubble nuclei are formed in supersaturated solutions and are grown by gas diffusion. In addition, no obvious crystalline particles were observed in the sample surface. The smooth and homogeneous surface except for a small amount of impurities in Figure 3 (e), which is consistent with the XRD results discussed in Figure 2.

Moreover, Figure 3 shows the SEM of different particle size samples. As can be seen that the particle size has little influence on the morphology change of the samples, and the XRD patterns shows that the effect of particle size on the vitrification of samples is lower than that of holding time. This may be due to the particle size range of this experiment, which has little effect on the vitrification process.
Figure 3. SEM section photographs of sample (a: 1100 ℃, 30 min, 200 mesh, b: 1150 ℃, 30 min, 150 mesh, c: 1200 ℃, 30 min, 100 mesh, d: 1250 ℃, 30 min, 80 mesh, e: 1300 ℃, 30 min, 60 mesh).

4. Conclusion
In this work, the best technology process of soil vitrification was created by combining orthogonal experiment design with microwave. Comparison with experimental results, the solidified body exhibits higher amorphization rate after being holding for 30 min at 1300 ℃ by microwave sintering. This provides an experimental basis for the treatment of contaminated soil by microwave technology.

Acknowledgments
This work was supported by the Doctor Foundation in Southwest University of Science and Technology (No. 10zx7126).

References
[1] Zhang S, Zhang J, Cheng X, et al. Electrokinetic remediation of soil containing Cr (VI) by photovoltaic solar panels and a DC-DC converter [J]. Journal of Chemical Technology and Biotechnology, 2015, 90(4): 693-700.
[2] Aguilar J, Dorronsoro C, Fernández E, et al. Soil pollution by a pyrite mine spill in Spain: evolution in time [J]. Environmental Pollution, 2004, 132(3): 395.
[3] Simón M, Martín F, Ortiz I, et al. Soil pollution by oxidation of tailings from toxic spill of a pyrite mine [J]. Science of the Total Environment, 2001, 279(1-3): 63-74.
[4] ASADA M, KAIMI E. Phytoremediation as a remediation technology of contaminated soil and ground water [J]. Journal of Environmental Conservation Engineering, 2005, 34(4): 264-271.
[5] Wang S, Mulligan C N. An evaluation of surfactant foam technology in remediation of contaminated soil [J]. Chemosphere, 2004, 57(9): 1079.
[6] Dellisanti F, Rossi P L, Valdrè G. Mineralogical and chemical characterization of Joule heated soil contaminated by ceramics industry sludge with high Pb contents [J]. International Journal of Mineral Processing, 2007, 83(3): 89-98.
[7] Blosi M, Dondi M, Albonetti S, et al. Microwave-assisted synthesis of Pr-ZrSiO 4, V-ZrSiO 4 and Cr–YAlO 3 ceramic pigments [J]. Journal of the European Ceramic Society, 2009, 29(14): 2951-2957.
[8] Thridandapani R R, Folgar C E, Folz D C, et al. Microwave sintering of 8mol% yttria-zirconia (8YZ): An inert matrix material for nuclear fuel applications [J]. Journal of Nuclear
Materials, 2009, 384(2): 153-157.

[9] Demirskyi D, Cheng J, Agrawal D, et al. Densification and grain growth during microwave sintering of titanium diboride [J]. Scripta Materialia, 2013, 69(8): 610-613.

[10] Lagashetty A, Havanoor V, Basavaraja S, et al. Microwave-assisted route for synthesis of nanosized metal oxides [J]. Science and Technology of Advanced Materials, 2007, 8(6): 484-493.

[11] Subramanian V, Burke W W, Zhu H, et al. Novel microwave synthesis of nanocrystalline SnO2 and its electrochemical properties [J]. The Journal of Physical Chemistry C, 2008, 112(12): 4550-4556.

[12] Bilecka I, Djerdj I, Niederberger M. One-minute synthesis of crystalline binary and ternary metal oxide nanoparticles [J]. Chemical Communications, 2008, 2008(7): 886-888.