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Fabrication of Calcined Clay Granule Comprising Zeolite

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This research tried to find out the optimum fabrication method of calcined clay granules comprising zeolite. Kaolin clay and natural zeolite powder were used as raw materials of calcined clay, and silica stone powder was used for controlling the porosity of the granules. The granulation was performed with two kinds of granulators: a pan granulator and a high-shear mixer granulator. Various granules were fabricated by the mixing ratios and the rotation speeds of the granulators, and were heated from 400 to 700℃ at 100℃ interval. The crushing strength, pore size distribution, and CEC of the granules were measured. The evaluation method for the resistance of granules to human treading was created and the tests were conducted at dry and wet conditions. The resistance and crushing strength improved in proportion to the rotation speed of the granulator and the heating temperature, but the CEC decreased. The pellet made by the pan granulator did not have the strength against treading upon heating to below 700℃, but the pellet made by the high-shear mixer granulator endured the treading test upon heating to over 500℃

Key words: Calcined clay, Granulation, Zeolite, Cation exchange capacity, Treading test

Introduction

Most Korean soils have sandy loam and coarse sandy loam. Those soils tend to speedily leach alkali components and become acidic. To improve the resulting low nutrient holding capacity and pH, calcareous minerals and fertilizers are being used. Because those characteristics cannot fundamentally revive soil, the addition of soil conditioner to increase CEC is required (Seo, 2000; Jin, 1995).

Several researches have been conducted on the use of zeolite with a high CEC as a soil conditioner, but those focused on only the composition of zeolite or on increasing the CEC of natural zeolite, etc. (Choi and Hur, 1993; Choi et al., 1993; Choi and Hur, 1995). However, even with such zeolite, it should be granulated (or pelletized) to prevent wash away, but few research works are found.

An example of the granulation of powders is calcined clay. It has been used as a soil conditioner, because it can secure the space among the granules for water drainage and, simultaneously, can keep water for plants in the inner pore. The calcined clay comprising zeolite also can increase CEC of the targeted soil.

The calcined clay granule is an aggregate of clay and has a lot of pore created among the clay particles. For the zeolite to be functional within the granule, the granule should have a large amount of open pores connected to inside of granule. Also, the granule should be heated for proper strength and stability at the risk of decreasing CEC. Therefore, the calcined clay granule comprising zeolite should be fabricated via a suitable granulation method and a heating method.

Granules as soil conditioner must not only be strong enough to be handled, but also be stable against to weathering or pressure when the granules are mixed with soil. Particularly, the resistance of granules to human treading should be examined. In this research, we present the evaluation method for the resistance of granules to human treading, according to which the resistance of existing soil and that of the granules fabricated in this research were appraised and compared.

Therefore, the objective of this research was to find out the optimum fabrication method of calcined clay comprising zeolite. The granules were fabricated by various mixing ratio of law materials, granulation method, and heating temperature. Variations of CEC, porosity, and resistance against treading according to the fabrication methods were investigated.

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Material and Methods

Materials Kaolin was used as the low material in the fabrication of calcined clay. It was considered suitable for calcined clay because its high plasticity makes it easy to granulate by mixing it with many minerals. It also showed a pH value of around 6-7 after heating, whereas many clay minerals have manifested weak alkali after roasting at the preparatory experiment.

A product of natural zeolite powder (Zeo300 by Rexem, Inc.) was used as functional material for CEC.

Pelletizing with only clays, zeolite and kaolin, can restrict the pore size created inside of the granules. Therefore, coarse powder of silica stone was mixed with them to change the porosity of the calcined clay and the size of the pores.

The components of each material were analyzed by XRF (X-ray fluorescence spectrometer, MXF 1700, SHIMADZU, Inc.), and the mineral components of each material was investigated by XRD (X-ray diffractometer, RU 200, Rigaku, Inc). The particle size was measured by a laser micron sizer (LMS 30, SEISHIN, Inc.).

Fabrication of Calcined Clay Zeolite was heated for 2 hours from 400℃ to 1,000℃ at 100℃ interval, after which the changes in the CEC according to each temperature were investigated and the scope of the heating temperature that revealed the functionality was grasped. The CEC was measured using Ammonium Replacement Method (Kjeldahl method).

A test material with a mixture of zeolite and kaolin at a 1:1 weight ratio and a material with a mixture of zeolite, silica stone, and kaolin at a 2:1:1 weight ratio were manufactured. These two kinds of test materials were granulated into the spherical pellet with a size of 10-20 mm, using a pan granulator and a high-shear mixer granulator, as shown in Fig. 1.

Granulators that can successively fabricate pellets can be largely divided into four types: the pan (drum) type, the high-shear mixer type, the fluidized bed type, and the extruder and spheronization type (Ramaker et al., 2007). Among these, the first two were used in this research after they were found to have been highly available at the field and to have endowed the pellet with suitable incipient strength.

The pan granulator puts in powder at a uniform speed on the pan that rotated at a slope and simultaneously sprays the binder on the powder. The particles that the binder grouped together moved outward via a centrifugal force and gravity, and rotated together with the pan. The pellets that reached the upper part fell toward the central part of the pan, grouped back together with the other particles, and repeated their rotation and fall. In such a process, a round pellet was formed and its strength generally depended on its weight, falling distance, etc. Besides, the characteristics of the pellets varied according to the size and the surface feature of particles, the ratio of the powder and the binder, the viscosity of the binder, the spraying position and dispersal degree of the binder, the speed of the input test material, the slant of the pan, the speed of the rotation, etc. Moreover, in many cases, these characteristics are controlled mainly with the operator's experience. In this research, only water was used as the binder for the fabrication of each pallet. Feeing rate of

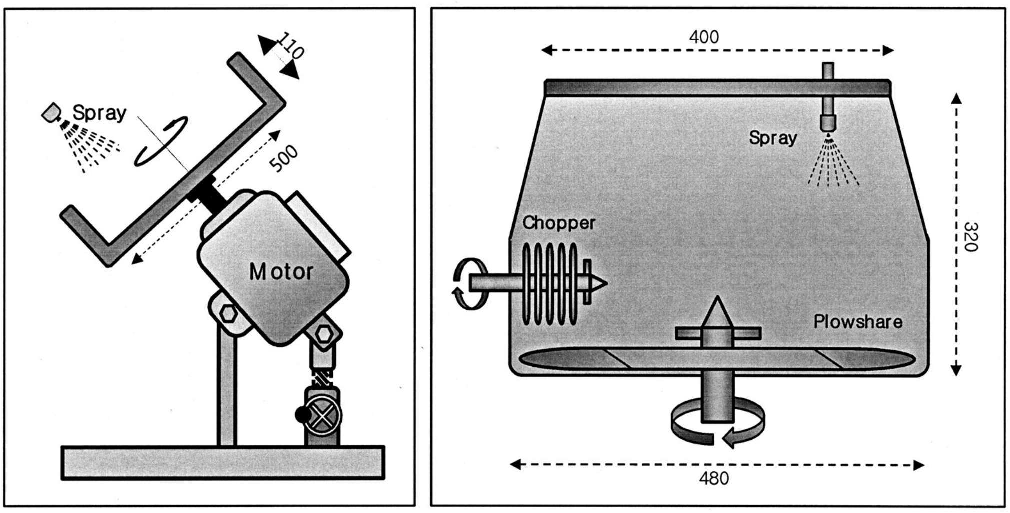

Fig. 1. Scheme of pan granulator (left) and high shear mixer granulator (right)

law materials was 3kg/hour and water content of the fabricated pellet was about 12~15%. The slant of the pan was fixed at 45℃ and with only the pan's rotation speed varied at 10, 20, and 30 RPM. The characteristics of the fabricated pellets were then compared.

The high-shear (HS) mixer granulator used in this research was the vertical axis type, in which the plowshare (impeller) that rotated horizontally in the lower part spun the powder on the vertical axis, and the chopper that rotated at the side blended the powder at the upper and lower parts, and dispersed the pellets properly that were condensed to excess. Concurrently, when the binder was sprayed from the upper part, the powder became granulated in their condensation and rotation. Although the powder, the nature of the binder, the rotation speed, etc. also affected the HS granulator, it was less affected by the incipient circumstances than the pan granulator, and thus facilitated the formulation of variables.

The strength of the pellets fabricated with the pan granulator depended much on the falling distance?i.e., on the size of the pan; whereas the strength of the pellet fabricated with the HS granulator depended on the rotation speed and the weight of the powder inside the granulator because it was affected by the centrifugal force created by the plowshare. In this research, the pellet was fabricated by constantly inputting 16 kg of the powder, regardless of its kind, at which time each pellet was fabricated with a different rotation speed of the plowshare at 60, 90, and 120 RPM and the characteristics of the fabricated pellets were compared.

The pellets in diverse circumstances were all made with a size of 1-2 cm. After they were dried, crushed with a cone crusher, and only the pellets sized 2-4 mm were classified and taken. This process endow the pellets with a lump shape, and it was required because the high flow ability of the pellets granulated in a round shape did not guarantee stability on a slope, slanted surface, etc.

Each pellet was heated for 2 hours from 400℃ to 700℃ at intervals of 100℃. The correlation between the fabrication circumstances and the functionality revelation was investigated by measuring the strength and the CEC.

Measurement of Strength The strength of the calcined clay granule was measured in reference to the KS standard KSEISO 4700, which measures the crushing strength of granules. This method measure the compressive force that can cause a crack in the granule. The granule is placed between two parallel plates, and a compressive force is applied by narrowing the gap between the plates at a constant speed. The maximum load at the time of the crushing is recorded as the crushing strength. In this research, the plates were moved at the speed of 40 mm/min and the crushing strength of the granule was measured by using a digital force gauge (FGP-20, NIDEC-SHIMPO, Inc.).

The value of the crushing strength is significantly affected by the size, shape, ratio of the length and the breadth, etc. of the granule (Bartake and Singh, 2007). The calcined clay granule used in this research was not uniformly shaped, and it reduces credibility of the measured value of the crushing strength. Besides, as there is no standard of suitable strength for soil, the usability of the measured value of the crushing strength must be lower.

Accordingly, an experiment (Fig. 2) was carried out to fabricate the calcined clay granules with the needed strength for soil in consideration of human treading. The test granule of 50 g was contained in a round-shaped mold with a 50 mm internal diameter, and human treading was continuously applied from the upper part using a piston. After a man whose weight was adjusted to 60 kg (by lifting a thing, etc.), the average weight of an adult, passed by and treaded on the appliance in Fig. 2 with 100 times, the degree of breakage of the material in the appliance was measured.

The size of the tested granules was 2-4 mm. The weight of the crushed granules was measured by sieving it with a 2mm sieve after the experiment. The crushing ratio was calculated as follows.

Crushing ratio (%) = (Amount of the test material sized less than 2 mm) / (Amount of the total test material) x 100

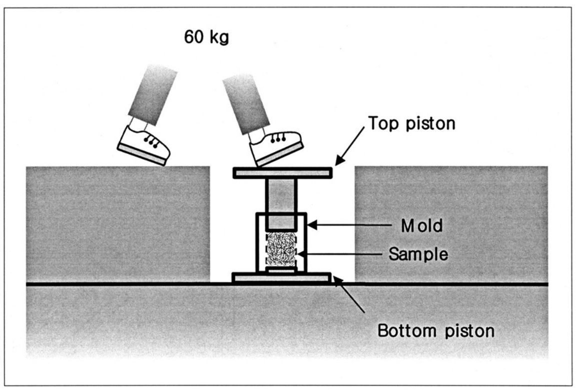

Fig. 2. Treading test for measuring strength of calcined clay.

If mixed with water and dried, clay solidifies and strengthens; but if again mixed with water, the solidified clay dissolves, and loses strength. Calcined clay is basically strengthened through sintering, but this clay can be considered to have a high degree of strength due to the strength created by the solidification of the clay. Therefore, the strength of calcined clay must be measured after it absorbs water and when it is dry. In this research, the crushing strength and resistance to human treading were measured with the granules that were dried and immersed in water for over 24 h, and the values were compared.

Results and Discussion The chemical and mineral compositions of the zeolite, kaolin, and silica stone were analyzed with an XRF and an XRD. The results are shown in Table 1 and Fig. 3. The main minerals of zeolite were mordenite and clinoptilolite, which typically represented the mineral component of natural zeolite in Korea.

The kaolin used in this research was the low-grade type from Sancheong, which has light pink color and contain a considerable amount of albite. Although 7?-halloysite and kaolinite with similar XRD peaks, judging from the preliminary observation in microscopy and the colors of mineral, the XRD peak in Fig. 3 is considered mostly for kaolinite. According to the studies that investigated about kaolin in Sanscheong area, the kaolin was formed by weathering of anorthositic rocks. It also reported that minerals that are more advanced with regard to weathering have more low-crystallized kaolinite and more color, and that weathering of biotite increase the content of Kaolinite in kaolin (Kim et al., 1989; Jeong and Kim, 1989; Jeong et al., 1995).

Fig. 4 shows the particle size of the three mineral powders used in this research. The average sizes of natural zeolite, kaolin and silica were 6.62㎛; 8.41 ㎛ and 74.83㎛, respectively.

The change in the CEC from the heating temperature of zeolite is shown in Fig. 5. It was found that the higher the temperature was, the less the CEC was. Especially in the over 800℃ temperature, the CEC radically decreased to under 50% of the original CEC and this result is similar to the experimental result that used artificial zeolite (Kim et al, 2007). Therefore, this research attempted to find a suitable heating temperature below 800℃.

The granulators used in this research were the pan granulator and the HS granulator. The characteristics of the granulators significantly influences to the characteristics of the granule. While the pan granulator compacted the granule by falling that on the pan, the HS granulator compacted the granules not only by the centrifugal force but also by load from the other granules

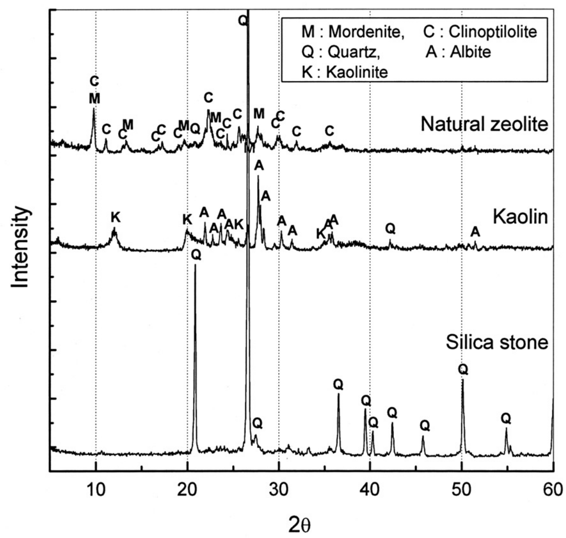

Fig. 3. X-ray diffraction patterns of samples (Cu Kα)

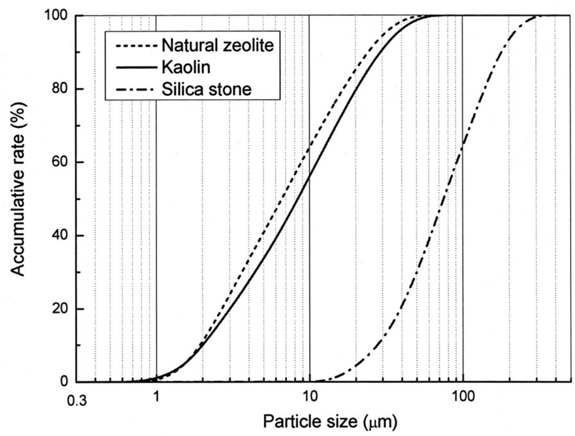

Fig. 4. Particle size distributions of samples.
(Mean size: natural zeolite: 6.62㎛, kaolin: 8.41㎛, silica stone: 74.83㎛)

Table 1. Chemical composition of samples analyzed by XRF (wt%).

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	TiO_2	MnO	P_2O_5	Ig. loss
Natural zeolite	67.22	13.67	2.45	1.87	1.08	2.49	2.16	0.35	0.08	0.06	8.32
Kaolin	49.29	32.68	1.43	5.69	0.47	0.34	1.64	0.18	0.02	0.02	8.32
Silica stone	95.05	1.32	0.83	0.01	0.01	0.03	0.02	0.02	0.01	0.01	2.26

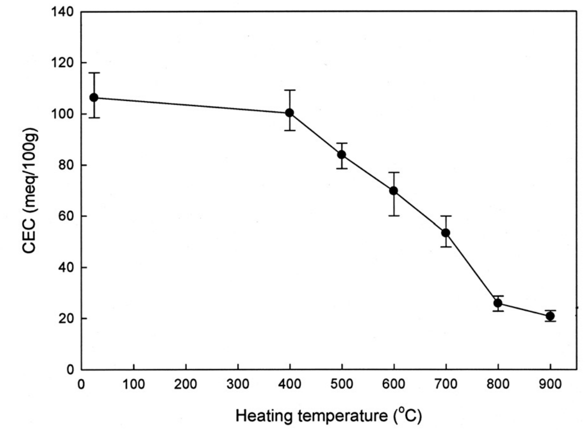

Fig. 5. CEC of natural zeolite at different heating temperature (heated for 2hours).

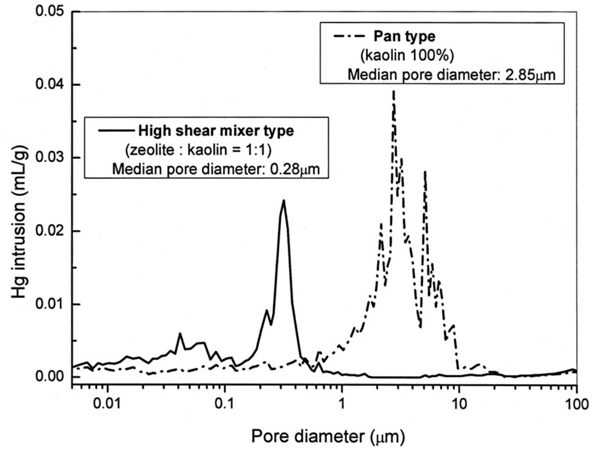

Fig. 6. Pore size distribution in granules made by two kinds of granulators (heated for 2 hours at 700℃)

inside the granulator (Salman et al., 2007). Therefore, the HS granulator has higher possibility to produce the granule with high strength.

Fig. 6 shows the distribution of the internal pores in the granules made by the two granulators. The granules made by the pan granulator had larger pores and higher porosity; the average sizes of the pores were 2.85㎛ and 0.28㎛, and the inside porosities were 58.5% and 37.49%, respectively. The rotating speed produced no significant difference on the pore characteristics and this result is similar to the research that investigated the characteristics of HS granulator (Schaefer et al., 1993).

Table 2 shows the crushing strength of the granules fabricated in various circumstances. Heating the granules made by the pan granulator at 400℃ did not produce enough strength even to maintain the form of that; and

Table 2. Crushing strength of granules made with different granulators and heated at different temperatures. (unit: kgf)

Sample	Test method	Heating temp.(℃)	Granulator and rotating speed					
			Pan granulator			High shear mixer granulator		
			10rpm	20rpm	30rpm	60rpm	90rpm	120rpm
Zeolite: kaolin = 1:1	Dry	400	N. D.	N. D.	N. D.	3.33 (3.39~4.61)	(3.19~3.73) 4.35	3.92 (4.14~4.56)
		600				4.55 (3.99~4.86)	4.67 (4.14~5.46)	4.65 (4.22~4.82)
		700	0.12 (0.10~0.16)	0.13 (0.11~0.14)	0.27 (0.24~0.28)	4.89 (4.60~5.11)	5.23 (4.26~5.85)	5.70 (4.92~6.55)
		1000	1.29 (1.06~1.68)	1.96 (1.74~2.20)	1.74 (1.43~1.80)			
	Wet	400				N. D.	N. D.	N. D.
		600				3.37 (2.92~4.38)	4.12 (3.27~5.24)	4.02 (3.46~4.74)
		700	N. D.	N. D.	N. D.	4.85 (3.42~6.79)	5.87 (4.85~7.12)	6.82 (5.77~7.40)
Zeolite: silica: kaolin = 2:1:1	Dry	600				3.79 (3.25~4.22)	3.53 (3.16~4.18)	4.22 (3.56~4.64)
		700	0.13 (0.09~0.14)	0.11 (0.10~0.13)	0.13 (0.10~0.15)	5.07 (3.79~5.40)	5.58 (4.64~6.56)	4.91 (4.14~5.84)
	Wet	600				2.82 (2.67~3.15)	3.26 (2.89~3.74)	3.70 (3.41~4.38)
		700	N. D.	N. D.	N. D.	3.82 (3.54~3.97)	3.34 (3.19~3.56)	5.57 (5.17~6.69)

N. D. : Because pellet did not maintain its shape, crushing strength was not detected.

the strength by heating at 1,000°C did not exceed 2 kgf. Even at 700°C, which was set as the maximum heating temperature in this study, the pellet could not maintain its shape in the wet state. Such lack of strength might be overcome by using a larger pan granulator, but this was impossible with the granulator used in this research. Therefore, in the following experiment, the granules were fabricated using only the HS granulator, and the characteristics were evaluated.

In the granules made with the HS granulator, it was appeared that the strength increased according to the heating temperature and the speed of rotations. The rotating speed is proportional to the input energy for granulation and it has influence on the strength of granules (Knight et al. 2000). All the granules showed the strength of over 3 kgf when dry, but the granules heated at 400°C did not show strength when wet. Therefore, the heating temperature must be around 500~700°C. The following experiments on resistance to human treading were conducted on the granules heated at 500°C and 700°C.

Fig. 7 shows the changes of the CEC with increasing the heating temperatures of the granules, which were fabricated at 60 and 120 rpm of rotation speed of the plowshare. The reduction in the CEC with increasing the temperature was marked, but only slight differences resulted from variations in the rotation speed. The granules of 2:1:1 mixture revealed a higher CEC value, which can be attributed to the contribution of large particles of silica stone to the security of the porosity. Moreover, since the blending ratio of zeolite for all the test materials was 50%, it can be seen that 50% of the measured CEC value was the maximum value of the granules (Fig. 5). The 2:1:1 granules would have had a CEC value of around 70-80% of the original test material, and the 1:1 granules would have had a CEC value of around 60-70% of the original in the heating at 400-600°C. Such results show that a certain part of the CEC is not exercised with granulation and that this can be improved by increasing the pores, such as by mixing the material with large particles.

Since the calcined clay granules used in this research were not uniformly shaped, the strength of the granules was investigated at simulated situation (Bartake and Singh, 2007). The strength test method considering human treading in Fig. 2 was prepared and conducted (Table 3). The lower the crushing ratio was, the higher the resistance to human treading was. As seen from the results, the granule of 1:1 mixture had a lower crushing ratio than that of 2:2:1. The dry granule of 1:1 showed crushing ratio of below 10% and the wetting of the granule hardly changed its crushing ratio except for that heated at 500°C. However, the wetting increased the crushing ratio of the 2:1:1 granule and the granule heated at 600°C showed almost 30% crushing ratio. Accordingly, the 2:2:1 granule proved good in terms of its use of the CEC of zeolite, but because it was less resistant to human treading, its granulation at a ratio of 1:1 and at a temperature of over 600°C can be regarded as good.

The above-mentioned results show that low heating temperature and rotation speed of granulator was beneficial for CEC, but harmful for the crushing strength and resistance to human treading. Thus, considering heating energy, human treading, CEC, etc., it is deemed proper to fabricate calcined clay granule that contains zeolite not by mixing coarse particle but by heating it at 600°C using an HS granulator. Fig. 8 shows a photograph of calcined clay granules fabricated in the above-mentioned conditions.

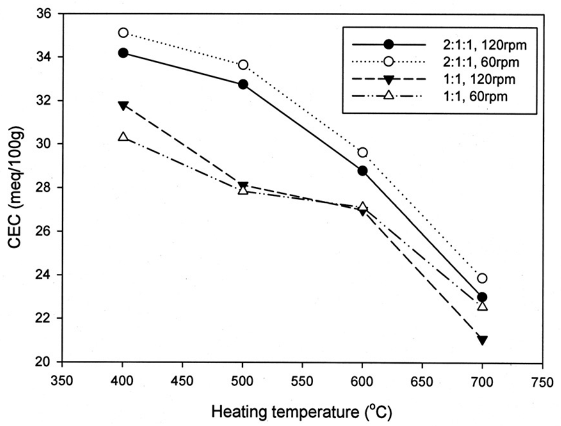

Fig. 7. CEC of granules made by high shear mixer granulator at different heating temperature (heated for 2hours).

Fig. 8. Picture of granule of calcined clay (2~4mm, zeolite:kaolin=1:1)

Table 3. Crushing ratio in treading test of granules. (unit: %)

Sample	Test method	Roasting temp.(℃)	Rotating speed of high shear mixer granulator		
			60rpm	90rpm	120rpm
Zeolite: kaolin = 1:1	Dry	500	10.44 (9.32~12.57)	-	9.41 (8.29~12.09)
		600	7.72 (7.18~8.78)	5.75 (5.08~6.32)	6.91 (6.26~7.56)
		700	6.88 (6.44~7.52)	5.48 (5.04~5.72)	6.52 (6.11~6.94)
	Wet	500	26.42 (19.14~31.47)	-	27.16 (24.68~30.05)
		600	8.76 (8.44~8.92)	8.42 (8.25~8.71)	9.21 (8.62~9.47)
		700	8.24 (7.53~8.48)	9.76 (9.35~10.02)	8.84 (8.42~9.30)
Zeolite: silica: kaolin = 2:1:1	Dry	600	8.83 (7.74~9.92)	9.67 (9.08~10.26)	7.65 (7.38~7.92)
		700	8.91 (8.68~9.14)	9.80 (8.34~11.26)	7.78 (7.37~8.21)
	Wet	600	28.42 (24.30~31.94)	25.90 (19.16~33.60)	17.53 (14.06~23.15)
		700	25.27 (19.61~27.04)	17.04 (14.26~21.11)	14.41 (10.28~16.80)

The experiment on the resistance to human treading was also applied to general soil: forest soil in the neighborhood of a mountain-climbing trail. The experiment was performed on only 2~4mm of sand collected by sieving of that. The crushing ratio all turned out to have been 15~25%. Such results may show that the calcined clay granules fabricated in this study was a soil conditioner suitable or mixture with existing soil. However the results of the experiment on human treading hardly reflected a small difference in the crushing strength. Also, because the crushing ratio is measured by sieving, small errors from sieving will be basically included. It is thus considered that henceforth, the test methods on human treading should be continually advanced through supplementation.

Summary

Suitable method of fabricating granule comprising zeolite for the use of a soil conditioner was investigated. Granules were fabricated by varying the heating temperature, the conditions of the granulator, and the mixing ratio of three powders such as zeolite, kaolin, and silica stone. To increase CEC of zeolite, granules must be heated under 700℃ and blended with silica with a large size to lower the reduction of the CEC from the granulation. The measurement of the crushing strength of the granules showed that the granules fabricated with a pan granulator were not strong enough, but those made with a high-shear mixer granulator showed enough strength after heating at over 500℃. The test method of granules to evaluate the resistance to human treading was designed and conducted at two situations: dry and wet. The granule made of 2:1:1 mixture of zeolite, kaolin, and coarse powder of silica stone showed the lack of resistance to human treading when wet. However the granules made of 1:1 mixture of kaolin and zeolite showed enough resistance when heated at over 600℃. Therefore, in the experimental conditions created in this research, it was found that granules can be suitably fabricated for the use of zeolite as a soil conditioner by granulating a mixture of zeolite and kaolin using a high-shear mixer granulator, and after drying the mixture, crushing and seiving it out by 2-4 mm, and heating it at a temperature of 600℃.

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제올라이트를 함유하는 소성점토의 제조

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제올라이트는 높은 양이온교환능력(CEC)으로 인해 토양개량제의 사용이 고려되어 왔으나, 이에 관한 연구들의 대부분이 제올라이트를 합성하거나 천연제올라이트의 CEC를 증가시키는데 국한되어왔다. 실제로 제올라이트를 토양개량제로 사용하기 위해서는 유실되는 것을 방지하기 위한 과립화 (granulation or pelletizing)가 요구된다. 본 연구는 제올라이트를 토양개량제로 활용하기 위해 이를 포함하는 소성점토의 과립 (calcined clay granule)을 제조하고, 제올라이트의 특성을 최대로 활용할 수 있는 과립의 제조조건을 찾으려 하였다. 천연제올라이트, 고령토, 규석을 재료로 하여, pan granulator와 high shear mixer granulator의 두가지 과립기를 사용하여 과립을 제조하였다. 재료의 혼합비와 과립기의 회전속도 등을 조절하여 과립을 제조하였으며, 이를 400~700℃범위에서 100℃단위로 소성한 후 파쇄강도 (crushing strength), 기공분포 (pore distribution), CEC를 측정하였다. 토양개량제로 사용할 과립은 취급시에 요구되는 기본적인 강도뿐만 아니라 토양과 혼합된 후 답압(踏壓)에 견디기 위한 강도 또한 요구된다. 본 연구에서는 과립의 답압에 관한 저항성 평가방법을 마련하고, 마른 상태와 젖은 상태에서 과립의 저항성을 측정하였다. 과립기의 회전속도와 가열온도에 비례하여 답압에 대한 저항성은 증가하나 CEC는 감소하였다. Pan granulator는 700℃이하의 가열에서 적절한 강도를 갖는 과립을 제조할 수 없었으나, high shear mixer granulator는 500℃이상의 가열에 의해 답압에 저항성을 갖는 과립을 제조할 수 있었다.