

# EFFECT OF YELLOW LOESS ON ODOROUS GAS ADSORPTION AND ECOTOXICITY TEST USING *DAPHNIA MAGNA*

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## ABSTRACT

To minimize damage caused by harmful substances released from artificial construction materials, there has been increasing interest in eco-friendly houses constructed with natural materials. Among natural materials, yellow loess is South Korea's most representative eco-friendly construction material, with high purification performance for indoor use. However, there has been no objective assessment of yellow loess's performance at purifying indoor air. In this study, internal pores were found in yellow loess that were of a lamellar crystal structure consisting of bonds of silica and alumina plates, and there was a high negative charge on the loess surface; these might have contributed to the excellent performance of yellow loess in adsorbing odorous gas. There was also an exposure test using *Daphnia magna* to determine the eco-friendliness of yellow loess compared with that of cement mortar. Results showed the survival of *Daphnia magna* was longer in the indoor environment of a test chamber with higher concentration of ammonia wherein yellow loess was placed. EC50 (median effective concentration, the concentration that caused death of 50% of bio-specimens) value of 19 ml of ammonia (0.1% solution), which was three times more than EC50 value of 6 ml of ammonia of the same area of cement mortar.

## KEYWORDS

eco-friendly material, yellow loess, *Daphnia magna* evaluation, CEC, gas adsorption

## INTRODUCTION

Recently, "passive houses" have become popular for their use of natural energy and efficient control of thermal energy (Rohdin et al. 2014, Figueiredo et al. 2016, Mihai et al. 2017, Tronchin et al. 2018, Tomas et al. 2018). To accomplish this high performance, thermal insulation is used in passive houses to keep occupants comfortable through indirect means and minimization of the need for energy-consuming equipment; direct equipment thus is only needed for limited essential purposes. However, excessive use of thermal insulators and the resulting reduction in ventilation for efficient control of thermal energy can deteriorate indoor

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air quality in passive homes. In particular, the air conditioning equipment that is the sole direct equipment in passive houses has limited capacity to prevent contamination of indoor air quality from sources such as concrete, furniture, and interior finishing materials. Many researchers have shown that concrete can emit  $\text{NH}_3$  (Kobayashi 2000, Jang et al. 2016, Bai et al. 2006, Puhakka et al. 2000); this gas is caused by the hydration of cement concrete (or mortar), and it can diffuse through concrete walls, resulting in increasing air pollution;  $\text{NH}_3$  is easily released in the high pH environment of concrete, and it is especially well-known as a highly toxic gas in the human body (Liang 2001).

Because of the above factors, the Korea Agency for Infrastructure Technology Advancement and other government agencies have been conducting research to identify methods of maintaining pleasant indoor air quality while maximizing energy consumption efficiency with active use of highly efficient thermal insulators and natural interior materials with high purification performance. For example, the Ministry of Land, Infrastructure and Transport is hosting a project aimed at constructing traditional Korean houses in the new style. The homes will be in the traditional Korean style but using construction materials with high energy efficiency and interior finishing materials such as yellow loess with highly effective purification performance and indoor humidity control for improved indoor air quality. The yellow loess is able to purify indoor air where direct air conditioning equipment cannot, and insulators reduce the consumption of thermal energy.

However, few researchers have explored the effectiveness of natural finishing materials such as yellow loess at improving indoor environments. Authors of most current studies in this context have focused on improving or maintaining indoor air quality or on emissions of hazardous materials from natural construction materials such as formaldehyde, total volatile organic compounds, and radon (Harb et al. 2018, Thevenet et al. 2018, Uwe et al. 2016, Hwanget al. 2017, Basma et al. 2018, Al-kahteb et al. 2017).

In the present study, we measured the physicochemical properties of yellow loess associated with polarized gas adsorption to identify loess's purification mechanism in indoor air. In particular, we analyzed the cation exchange capacity (CEC) of yellow loess using neutral ammonium acetate to identify the relevance of loess's surface charge to polarized gas adsorption. In addition, we used a *Daphnia magna* species known to react to chemical substances sensitively as a bio-specimen for ecotoxicity testing to evaluate the polarized gas adsorption of yellow loess by evaluating the growth of *D. magna* exposed to ammonia gas at predetermined durations.

## MATERIALS AND METHODS

### Materials

Specimens of yellow loess were obtained from Hongcheon, Iksan, Gochang, and Sancheong, representative yellow loess production areas in South Korea. They were dried at  $105 \pm 5^\circ\text{C}$ , crushed, and sieved with no. 80 mesh (mesh size  $180 \mu\text{m}$ ) for the analysis of raw specimens (Table 1).

### Measuring CEC

Neutral ammonium acetate has been employed in previous studies (Taylor 1958, Hanlon et al. 1984). For the present study, we used the experimental method presented in "Soil and Plant Analysis II—Chemical Properties of Soil" (Yang, 2003) published by the National Institute of Forest Science. The neutral ammonium acetate method measures Mg, Ca, K, and Na exchanged

**TABLE 1.** Chemical properties of general soil and yellow loess powder.

Sample	Oxide composition (%)											
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	P <sub>2</sub> O <sub>5</sub>	TiO <sub>2</sub>	MnO	K <sub>2</sub> O	CaO	Na <sub>2</sub> O	MgO	BaO	SrO
Soil	34.94	16.84	0.54	0.41	0.52	0.26	0.54	38.8	0.52	4.7	0.12	0.08
Hongcheon	66.8	20.1	6.65	0.103	1.16	0.106	2.83	0.241	0.26	1.66	—	—
Iksan	58.7	31	5.47	—	1	—	3.45	0.133	0.108	—	0.108	—
Gochang	59.1	26.8	8.49	0.151	1.27	—	2.99	0.158	0.138	0.913	—	—
Sancheong	51.4	40.4	6.61	—	0.358	—	0.589	0.243	—	0.436	—	—

with saturated ammonium ion (NH<sup>+</sup>). To prepare a solution of 1N-NH<sub>4</sub>OAc, 77.1g of NH<sub>4</sub>OAc was added to a flask of 1,000 ml filled with 900 ml of distilled water. The pH of the solution was adjusted to 7.0 by applying a solution of 3N-CH<sub>3</sub>COOH or 3N-NH<sub>4</sub>OH, and the remaining volume of the flask was filled with distilled water up to 1,000 ml. After 20 ml of extracting reagent was added to a conical tube containing 5g of specimen, the tube was agitated for 5 minutes in a reciprocating agitator. Thereafter, the conical tube was centrifuged at 3,000 rpm for 10 minutes, and the supernatant was removed from the conical tube. We then analyzed the Mg, Na, K, and Ca present in the supernatant with ICP-OES equipment.

### Preparation of Test Specimens

Cement mortar specimens were prepared with a water:cement ratio of 1:2 and a cement:sand ratio of 1:2.45. Specimens of yellow loess block, a mixture of water and yellow loess powder, were cured and dried completely in a mold (8 cm × 8 cm × 1 cm). Surfaces of all prepared specimens were sealed with aluminum tape except for an area of 6 cm × 6 cm that was left for all specimens to obtain equal test exposure areas.

### Evaluating Purification Performance for Odorous Gas

A representative odorous polarized gas (0.1% ammonia) was injected through the inlet of a sealed test chamber 5 l in volume, and each specimen was placed in the chamber; the chamber was held at a constant temperature of 20 ± 1°C. A detector tube was used to measure concentrations of injected gas. We assessed the gas purification performance of each specimen as the difference between the initial concentration of injected ammonia gas and that after a predetermined period of time.

### Test of Survival of *D. Magna*

*Daphnia magna* is a species of the genus *Daphnia* belonging to the family Daphniidae, order Cladocera of Diplostraca under phylum Arthropoda; it is used as an international standard species to study toxicities of various materials to aquatic invertebrate animals due to its high sensitivity to toxic compounds; a vast database on toxic materials and toxicity has been accumulated for this species. Due to its strong breeding potential, *D. magna* allows for easy indoor culture, and it reacts sensitively to chemical substances; the U.S. Environmental Protection Agency and countries around the world have employed *D. magna* to study chemical substances. In the present study, we used *D. magna* to conduct the EPA's ecotoxicity test (acute toxicity and fertility; EPA 2002). *D. magna* can be affected by airborne hazardous substances dissolved

in water and lose its mobility, and ammonia gas dissolves easily in distilled water. Thus, we selected ammonia gas from among diverse hazardous substances and injected it into the test chamber containing specimens of yellow loess and cement mortar plates or blocks to evaluate the survival of *D. magna* and determine the specimens' performance at purifying ammonia gas.

### Cultivating *D. Magna*

For this study, we used *D. magna* obtained from the Korea Institute of Toxicology (Daejeon Metropolitan City, Korea) and cultured according to EPA standards in an incubator at  $20 \pm 1^\circ\text{C}$  with 16 hours of light and 8 hours of darkness. About 45–50 individuals were cultured in each 2ℓ beaker filled with 1.8ℓ of hard reconstituted water (HRW). HRW (1ℓ) was prepared using the following chemical substances: 0.12g  $\text{MgSO}_4$  (magnesium sulfate), 0.192g  $\text{NaHCO}_3$  (sodium hydrogen carbonate), 0.008g KCl (potassium chloride), and 0.12g  $\text{CaCO}_3$  (calcium carbonate). The chemicals were mixed with deionized distilled water, and the mixtures were aerated for at least 24 hours before each test. The pH of HRW was maintained in the range of  $8.2 \pm 0.2$ . The *D. magna* culture medium was replaced with new HRW every other day. The *D. magna* was fed *Chlorella vulgaris* algae purchased from Aquatic BioSystems, Inc. (Ft. Collins, CO, USA) and YTC (a mixture of yeast, trout chow, and carophyll).

### Culture Chamber and Breeding Process

For the tests we conducted for the present study, indoor temperature and relative humidity were kept constant at  $20 \pm 1^\circ\text{C}$  and  $45 \pm 5\%$ , respectively. The common area of  $36\text{ cm}^2$  of each yellow loess-cement mortar block specimen (representative area: Gochang) was exposed to ambient space in a 7ℓ test chamber made of stainless steel into which ammonia water (0.1%) was injected to evaluate the purification performance of each specimen (Table 2, Figures 1–2). Fetal *D. magna* hatched from an egg of healthy adult *D. magna* after incubation up to 24 hours were collected for every test conducted in the present study; for each test, we tested a set of three beakers containing *D. magna*; each 50 ml beaker contained 10 individuals, and we measured the survival of the *D. magna* individuals in each specimen. We divided the experiment into two

**TABLE 2.** *Daphnia magna* test condition.

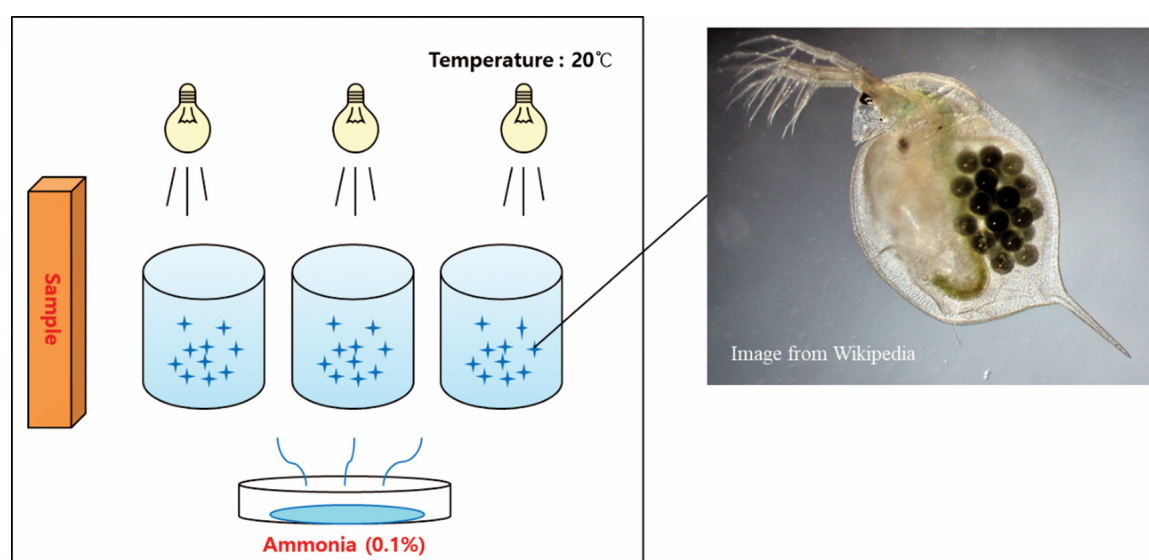
Test living thing	<i>Daphnia magna</i>
Living thing age	Less than 24 hours old
Living thing condition	1hour starvation condition before experiment
Chamber volume	7ℓ
Chamber temperature	$20 \pm 1^\circ\text{C}$
Intensity of light	14 LUX
Light cycle	16hour light, 8hour darkness
Experiment time	24hour, 48hour
number of repetitions	3 (expressed as mean $\pm$ standard error)

groups by exposure, either 24 or 48 hours; furthermore, all test organisms spent one hour in starvation before exposure until the results were confirmed.

### Judging *D. Magna* Survival

We judged whether the *D. magna* were dead or immobilized as follows. *D. magna* was judged dead from an observation of if we observed no reaction after 15 seconds of soft shaking of each test beaker, we judged that the specimens were dead. If we detected no motion of partial organs (such as feeler or post-abdomen) or no swimming we judged the *D. magna* to be immobilized; we also considered motion of feelers without swimming movement to be immobilization. We express the test results as the EC50 of each specimen, that is, the concentration that caused the death or immobilization of 50% of the *D. magna*, in accordance with EPA guidelines.

**FIGURE 1.** Diagram of experiment chamber inside.



**FIGURE 2.** *Daphnia magna* exposed to odorous gas.



(a) Yellow loess

(b) Cement mortar

(c) Breeding of *Daphnia magna*

## RESULTS AND DISCUSSION

### Analysis of Microstructure

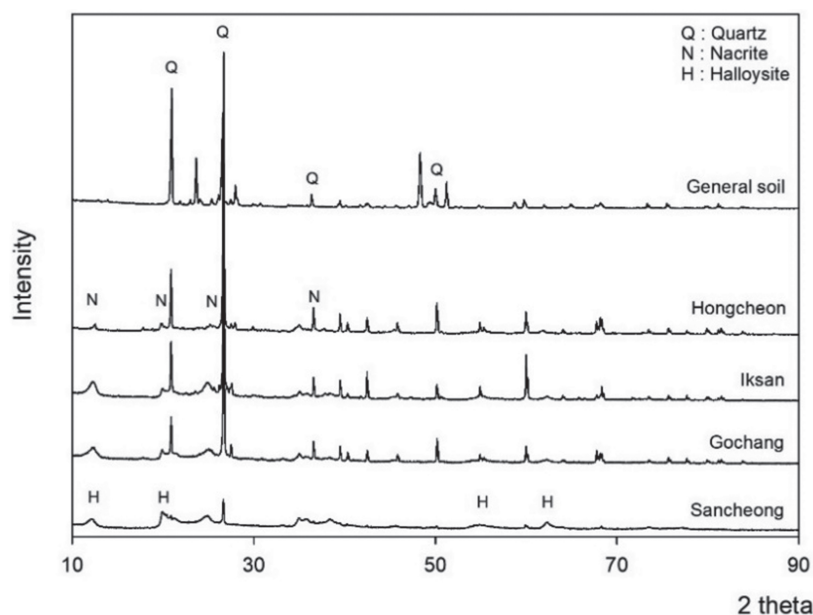
Yellow loess belongs to the kaolin clay family; the parent material of clay comprises the oxides  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{FeO}$ ,  $\text{MgO}$ ,  $\text{CaO}$ ,  $\text{Na}_2\text{O}$ , and  $\text{K}_2\text{O}$  originating from rock where  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  are dominant; thus, clay consists of substances resulting from decomposition or deterioration of rock (Mun 1996, Choi 2002, Cho 2015). We found chemical substances comprising  $\text{SiO}_2$  (51–66%),  $\text{Al}_2\text{O}_3$  (20–40%),  $\text{Fe}_2\text{O}_3$  (5–10%),  $\text{MgO}$  (0.4–2.5%),  $\text{CaO}$  (0.1–2.5%), and  $\text{K}_2\text{O}$  (0.5–4.1%) in yellow loess specimens taken from four locations, with  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{Fe}_2\text{O}_3$  dominant in these specimens. Their contents varied slightly by place, and we attributed these variations in Si and Al content to configurations of silicate tetrahedron and alumina octahedron in the clay minerals.

X-ray diffraction distinguished quartz, mica, feldspar, and clay minerals in the specimens from each location, including high peaks of quartz; these findings are presented in Figure 3.  $\text{SiO}_2$  has been found in sand and quartz distributed over a wide area (So, 2013). Nacrite, halloysite, and kaolinite are kaolin clay minerals, and we also commonly found these in the yellow loess specimens sampled from all four places. These clay minerals are of lamellar structure with covalent bonds between silicate tetrahedron and three atomic oxygen or alumina octahedron and hydroxyl groups. This crystal structure of clay minerals is characterized by a wider specific surface area owing to forms of either tube or between layers. The structure shows the characteristic of adsorbing water or contaminants into the inside of particles (Lee 2003, Kim et al. 2000).

### CEC of Yellow Loess

Clay takes a permanent negative charge by isomorphous substitution and edge charge. The permanent negative charge of kaolinite, a clay mineral of 1:1 lattice structure, comes from edge charge; it is extricated by the cut-off of bond at arbitrary points over the a or c axis of plate

**FIGURE 3.** X-ray differential patterns of yellow loess from different locations.



**TABLE 3.** Effective cation exchange capacity (ECEC) of soil and yellow loess.

Sample	Cation exchange capacity (cmol/kg)				
	Mg	Ca	K	Na	ECEC
Soil	0	3.1368	0.0664	0.0209	<b>3.2241</b>
Hongcheon	4.2039	5.4367	0.0053	0.0552	<b>9.7012</b>
Iksan	3.0772	5.0727	0.0061	0.0097	<b>8.1657</b>
Gochang	0.8437	15.0439	0.0125	0.0769	<b>15.977</b>
Sancheong	7.4752	16.1619	0.0115	2.4663	<b>26.1148</b>

crystal. Because edge charge is created only on the edge of clay minerals, increasing fineness by crushing clay minerals will increase the amount of negative charge, thereby increasing cation exchange capacity (Lee 2006).

Unstable negative charges distributed on the surfaces of yellow loess are stabilized through atomic bonds with diverse cations and water dipoles. Negative charges on the surfaces of the clay particles and the surface layer, which is referred to as adsorbent, can create electricity together, thereby pulling water dipoles and cations. Cation concentration in the adsorbent decreases as it becomes distant from surface of yellow loess, and this phenomenon of adsorption explains diverse features of clay such as cohesion force, lower permeability, and swelling shrinkage.

Measurements of CEC obtained from ICP-OES are presented in Table 3; we converted these measurements to units of cmol/kg or meq/100g before calculating the following equations. For Ca and Mg, we multiplied the converted measurement by equivalent weight of 2 in Equations (1) and (2). For K and Na, we divided the new measurements by their respective atomic weights because the equivalent weights in Equations (3) and (4) were 1. Equation (5) will render effective cation-exchange capacity (ECEC) through adding CECs of each element. Converting with neutral ammonium acetate gave CECs of 36 cmol/kg from specimens of yellow loess less than 0.3 mm of Gochang, while we obtained CECs of 27 cmol/kg and 19 cmol/kg for specimens from Sancheong and Iksan. The CEC of cement mortar was 0 cmol/kg.

$$\text{Exch. Ca} = \frac{(a-b) \times 20}{10 \times 20.04} \quad [\text{cmol/kg}] \quad (1)$$

$$\text{Exch. Mg} = \frac{(a-b) \times 20}{10 \times 12.15} \quad [\text{cmol/kg}] \quad (2)$$

$$\text{Exch. K} = \frac{(a-b) \times 20}{10 \times 39.10} \quad [\text{cmol/kg}] \quad (3)$$

$$\text{Exch. Na} = \frac{(a-b) \times 20}{10 \times 24.31} \quad [\text{cmol/kg}] \quad (4)$$

where  $a$  = mg/kg Ca, Mg, K or Na in the extraction solution,  $b$  = ditto in the blanks, 20 = ml of  $\text{NH}_4\text{OAc}$  used in extraction (the atomic weights were Ca = 40.078; Mg = 24.305; K = 39.098; Na = 24.305).

$$\text{ECEC} = \text{Exch.}(\text{Na} + \text{K} + \text{Ca} + \text{Mg}) \quad [\text{cmol/kg}] \quad (5)$$

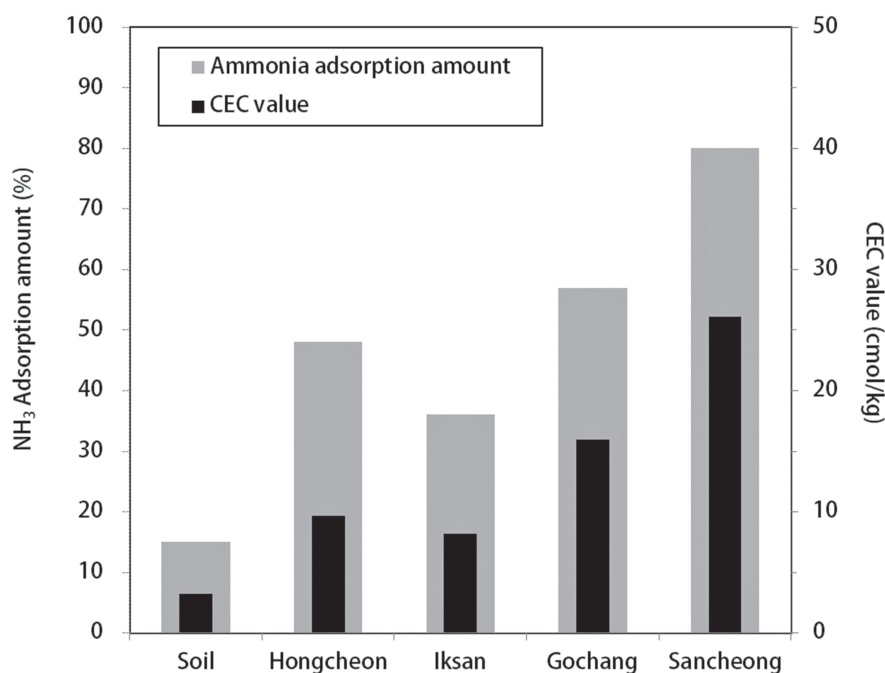
### Evaluating the Purification Performance for Odorous Gas

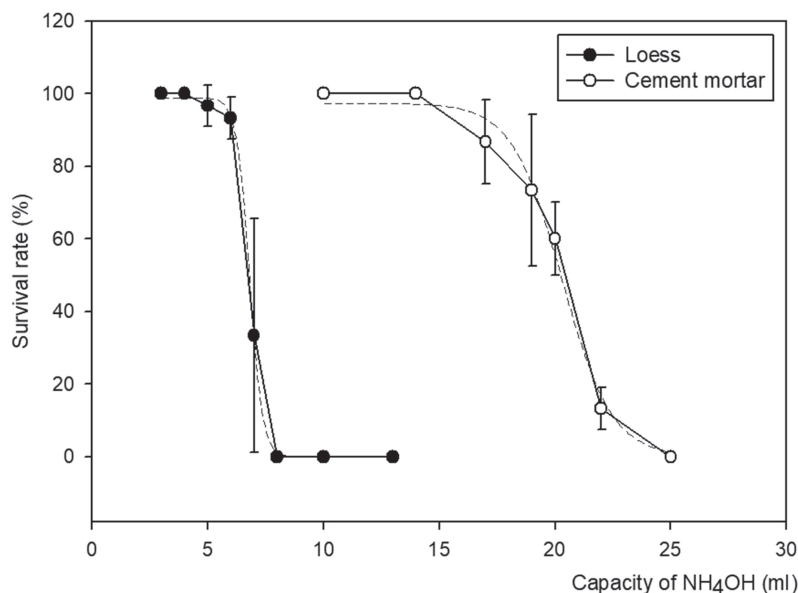
With the present study, we intended to identify correlations between CEC and ammonia gas adsorption capacity using the CECs of yellow loess specimens from each of four locations in Korea and the ammonia gas adsorption over time. We compared the amount of ammonia gas adsorbed for 6 hours in a chamber and the CECs of the yellow loess specimens as well as general soil; Figure 4 presents these results, which revealed a proportional relationship between CECs and ammonia gas adsorption capacity. Ammonia adsorption in yellow loess is known to be associated with the distribution of fine pores in yellow loess and its wide specific surface area. However, based on the abovementioned CEC results, we found that the negative charges on the yellow loess surfaces were critical for the adsorption. That is, yellow loess attracts gaseous particles in the cationic state with negative charges created on its surface as clay mineralizes.

### D. Magna Survival Rate

The test for exposing *D. magna* to an indoor environment contaminated with odorous ammonia gas was designed to evaluate how well the prepared specimens purified the gas and to determine the controllability of specimens for the growth of live organisms of *D. magna*. We prepared the 7ℓ test chamber by placing the prepared specimens (exposure area of 36 cm<sup>2</sup>), source of

**FIGURE 4.** Cation exchange capacity and  $\text{NH}_3$  adsorption amount of yellow loess.



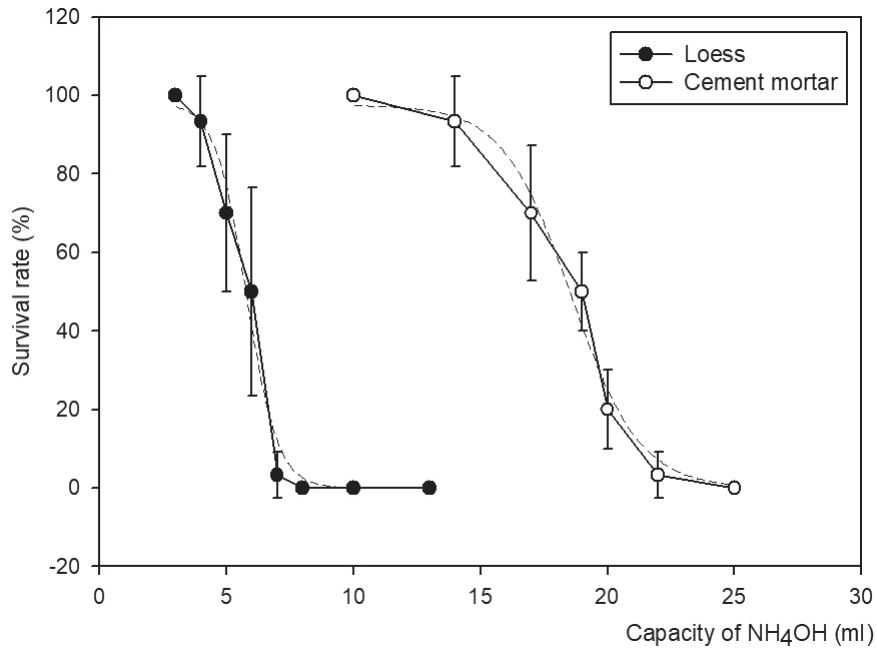
**FIGURE 5.** Comparison of 24-hr survival rate of *D. magna* with best fit curve.

ammonia gas, and repeated test groups of *D. magna* in the chamber to observe the state of *D. magna* for 24 and 48 hours. Our observation results are shown below; in particular, Figure 5 graphically depicts survival of *D. magna* exposed to ammonia gas for 24 hours in the chamber as average rate of survival plotted with standard deviations.

In the interval from 6 to 8 ml of injected ammonia solution in the cement mortar chamber, we observed a rapid decrease in *D. magna*'s survival rate; with 7 ml of ammonia solution injected, there was a significantly large standard deviation of 32.1455. In the interval, fine variation in ammonia gas concentrations appeared to critically change *D. magna* survival, but we observed a rapid decrease in the interval from 17 to 22 ml of injected ammonia solution in the yellow loess chamber. With the application of a 0.1% ammonia solution to the test chamber, the *D. magna* survival rate ranged from 0 to 100% corresponding to the following amounts of injected ammonia solution: 3–8 ml (cement mortar) and 14–25 ml (yellow loess). The *D. magna* survival rates in the yellow loess specimens showed a broader range than that of cement mortar. This suggests that yellow loess placed in the test chamber had superior ammonia gas adsorption to that cement mortar, and in fact, the *D. magna* survival curve corresponding to the cement mortar specimens was nearly a straight line, suggesting that cement mortar has no capacity for ammonia gas adsorption.

Figure 6 shows survival rates of *D. magna* exposed to ammonia gas for 48 hours in the test chamber; rates tended to decrease slowly in the interval of 4–6 ml of injected ammonia solution: 93% at 4 ml, 70% at 5 ml, and 50% at 6 ml in the cement mortar chamber. In the interval of 6–7 ml, the survival rate rapidly decreased from 50% to 3%, and at 6 ml of injected ammonia solution, the rate had a large standard deviation of 26.4575. We found that the survival rates of *D. magna* exposed for 48 hours were more desirable for determining the concentrations of ammonia solution that affected *D. magna* survival. As we noted above, the *D. magna* survival rate curve that corresponded to the cement mortar test specimen was more of a straight line, suggesting that cement mortar did not adsorb irrespective of the duration of time for adsorption. With the injection of 10 ml of 0.1% ammonia solution, 100% of *D. magna* survived in

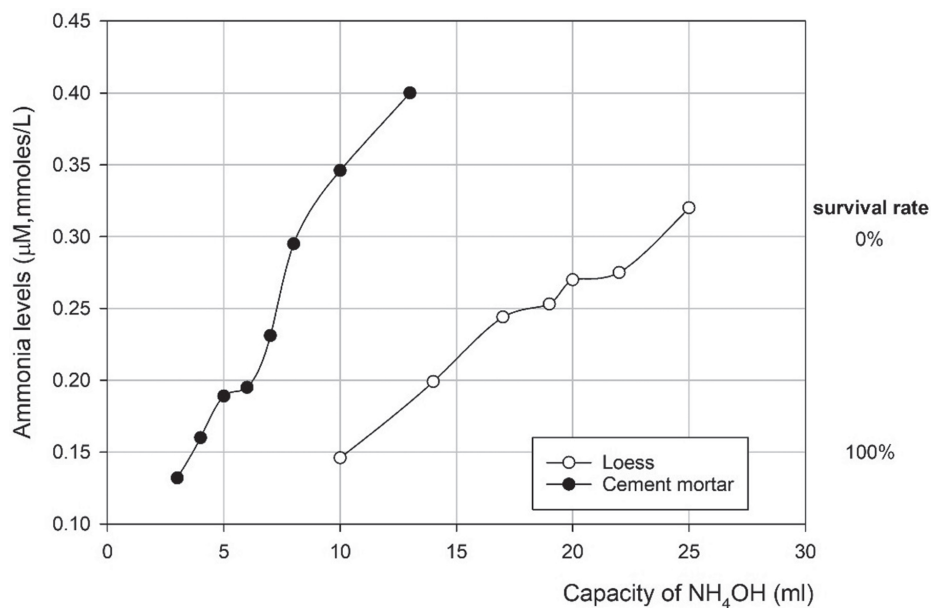
**FIGURE 6.** Comparison of 48-hr survival rate of *D. magna* with best fit curve.



the test specimen of yellow loess block, whereas 0% survived in the cement mortar. After 48 hours of exposure to the yellow loess test specimen, the EC50 was 19 ml, but under identical condition, it was 6 ml of ammonia solution for the cement mortar test specimen.

Figure 7 shows the concentrations of ammonia dissolved in distilled water injected into the test chamber for each test specimen followed by the observed ammonia gas adsorption

**FIGURE 7.** Absorbed ammonia levels in distilled water.



capabilities for 48 hours. When the ammonia concentration was over 0.3  $\mu\text{M}$ , the corresponding survival rate of *D. magna* was 0%, whereas when the concentration was below 0.15  $\mu\text{M}$ , *D. magna*'s survival rate was 100%. After 10 ml of ammonia solution was injected, the ammonia concentration in the yellow loess block test specimen was more than twice that in the cement mortar test specimen, demonstrating superior ammonia gas adsorption by yellow loess to that by cement mortar, similar to the results of the previous test.

## CONCLUSION AND RECOMMENDATIONS

In the present study, we conducted U.S. EPA ecotoxicity testing using *D. magna* to determine the eco-friendliness of yellow loess compared with that of cement mortar. Results are summarized below:

- *D. magna*, known to react sensitively to chemical substances, was employed as a bio-specimen for the ecotoxicity testing conducted in the present study. The EC50 for a 36  $\text{cm}^2$  area of yellow loess block was 19 ml in 0.1% ammonia solution, more than three times the EC50 of 6 ml for the same area of cement mortar in the same concentration of ammonia.
- Test results also showed that *D. magna* survived longer in an indoor test chamber at higher ammonia concentration gas when the yellow loess block was placed. Thus, yellow loess can be used in closed environments such as passive houses as an excellent means to reduce the use of air conditioning and purify or improve indoor air quality.
- For this study, we carried out an ecological experiment using water fleas to test the ammonia adsorption of loess. However, in order to more objectively evaluate environmentally friendly materials, it is necessary to accurately evaluate the complex adsorption performance of loess against harmful gases such as volatile organic compounds, formaldehyde, and radon that can occur indoors.

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