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The correlation between surface species on the heating material and heating performance for improving dry efficiency in thermal sewage sludge process

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ABSTRACT

Various metal oxides were added to prepare an exothermic heating material for microwave assisted sludge drying system. The highest heating performance was found when 10 wt% nickel was added to $LiNO_3$:6Fe_2O_3:ZnO(molar ratio). Thermal performance and reproducibility were improved when $Li_5Fe_5O_8$ and $NiFe_2O_4$ were formed on the surface of the heating material. During sludge drying process using exothermic heating block, the moisture content in sludge was reduced to under 1% after 5 min. By applying Li-Fe-Zn based heating material during the microwave drying, a drying efficiency of 43% was achieved.

1. Introduction

Dehydration and drying process must be preceded in order to efficiently treat sewage sludge with high moisture content. With The result of significant reduction in volume and mass of the sewage sludge by thermal drying process, substantial reduction of labor and transportation costs can be achieved, which are typically over half of the total cost for waste disposal. Also, It helps to minimize the damage to the environment by preventing leachate. The conventional sludge drying process is carried out hot air drying technology, which currently accounts for about 70% of the total drying technology [1,2] due to its simplicity of drying device and operation. However, heat dissipation to unwanted direction due to difficulties on heat flux control results in low drying efficiency and consequently high energy consumption with longer drying time during the process [1,3]. Furthermore, because of the uneven distribution of moisture content inside of the sludge, often excessive drying process is required to ensure sufficient degree of moisture removal [4,5]. On the other hand, low temperature drying process, typically below 60 ℃, also has been widely used for reducing moisture content while avoiding high energy consumption. However, the low temperature method demand much longer drying period due to slow evaporation of moisture. In addition, the process often suffers from poor quality of result caused by moisture gradient through the thickness of the sludge. It is a common issue because internal moisture content is extremely difficult to remove.

Recently, a microwave-assisted moisture removal technique draw attention for overcoming the challenges [2,3,6–13]. The level of energy consumptions of various technology including hot air drying and microwave drying have been reported in previous works. According to work by various research, the hybrid system of microwave drying and hot air drying system increases energy conservation and thermal efficiency compared to that of hot-air drying process [2,14–17]. The technique utilized the conversion of electromagnetic energy into thermal energy, and the thermal efficiency depends on the magnetic properties of the material [6,18–21].

The magnetic materials such as iron, nickel and cobalt typically generate heat when exposed to microwave. The thermal behavior of these materials under microwave exposure is affected by both electric field and magnetic fields. The electric field imparts motion to the free electrons, whereas the magnetic field affects the electron spin, domain wall and orientation of domains. The heat loss mechanisms in magnetic materials exhibit conduction losses with additional magnetic losses such

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as hysteresis, eddy current, domain wall resonance and electron spin resonance [22]. Silicon carbide (SiC), activated carbon, molybdate, nickel, cobalt, magnetite and ferric oxide have been reported as microwave-sensitive materials [23,24]. To improve the microwave heating efficiency, researches have reported on mixing various absorbing materials such as charcoal, graphite or nickel to SiC and zirconia [25,26]. Also a family of iron oxide such as Fe_2O_3 or Fe_3O_4 is one of the popular substances for generating heat because of their high sensitivity to microwave. It was reported that when Fe_2O_3 was reduced to FeO or Fe, the heating performance deteriorated. However, neither the modification of the Fe_2O_3 powder nor the detail of heating material fabrication process have been fully reported. Li and Zn, are known to be able to produce microwave-sensitive materials when alloyed with Fe. Lee et al. produced a microwave-sensitive heating material by mixing various metal oxides such as Fe_2O_3 for drying sewage sludge when the heating material was applied by microwave process, the sludge with 55% water content was discharged resulting in dry sludge with 1.7% water content [27]. In addition, Jang et al. and Lee reported that heating performance could be improved when $Li_5Fe_5O_8$ species was formed on the surface of heating material [28]. However, additional studies on stability, durability, and heating reproducibility are needed to utilize such heating material for industrial scale drying processes.

Therefore, in this study, to improve the performance and stability of heat material magnetic sensitive materials were added to fabricate a heating material and their heating performance were characterized. The surface properties were characterized by XRD analysis in order to correlate the exothermic performance to transform of the surface.

2. Material and methods

2.1. Materials preparation and characterization

2.1.1. Reagent information

In this study, an experiment was conducted to select optimal additives in order for the maximized performance and stability of the heating material. Disk shape heating blocks with different additives were prepared for the drying process. The reagents used in this work were summarized in Table 1.

2.1.2. Preparation of microwave-sensitive heating material (disk)

As previous study by Lee suggested [27], Lithium: Zinc: Iron oxide metal oxides were mixed in a 1: 1: 6 molar ratio using a mortar bowl for fabrication of the heating disk. 10% of lignin by weight was added as a binder to increase the mechanical stability [28]. Silicon carbide, nickel, and activated carbon, which are known to be microwave-sensitive materials, were added to the prepared powder at ranges of 10 wt%. Lastly, 3 g of the material mixture was molded at 6800 kg_f compression using a 1-in diameter disk mould. The melting point of Fe_2O_3 is 1565 °C and 750 °C or above is required for sintering to occur. According to Lee et al. porosity were measured to 2.6, 0.122 and 0.073 m^2/g when heat treated at 900 °C, 1200 °C and 1400 °C [27] respectively. In order to increase the thermal conductivity, the heating material have to prepared low porosity. So, to reduce a porosity and to improve the exothermic temperature, the mold was maintained at 1400 °C for 4 h in a nitrogen atmosphere then the materials were kept at 1400 °C for 4 h in air atmosphere. The heating materials prepared are listed in Table 2.

Table 1
Reagent information used to prepare the heating material.

Reagents	Chemical formula	Purity	Company
Lithium nitrate	$LiNO_3$	0.99	Sigma Aldrich
Iron oxide(III)	Fe_2O_3	0.95	Sigma Aldrich
Zinc oxide	ZnO	0.99	Sigma Aldrich
Lignin	$C_{18}H_{24}O_{11}\sim C_{40}H_{45}O_{18}$	–	Sigma Aldrich
Silicon carbide	SiC	0.975	Sigma Aldrich
Carbon	C	0.9999	Duksan chemical
Nickel nitrate hexahydrate	$Ni(NO_3)_2$-$6H_2O$	0.99	Sigma Aldrich
Nickel	Ni	0.97	Sigma Aldrich

2.1.3. Activity measurement

Fig. 1 shows the schematic diagram of the set up used for heating the heating material prepared in the study. The experiments were carried out at a low power of 500 W in order to reduce the risk of arcing during the microwave experiments [29]. The microwave oven was controlled by independent power supply (POWERSUPPLY Co., MGT-1100S) and temperature was monitored through a thermocouple (TENMARS, TM747DU). The Microwave oven was operated at a frequency of 2.45 GHz. The turntable in the microwave oven was rotated during the experiment to ensure uniform heating. In addition, the distance between the thermocouple and the samples were kept at 6 cm by an alumina tray sample holder on the turntable which is inert to the microwave that were used during the experiment. The temperature changes were measured using a K-type thermocouple on the randomly selected spots on the surface of the heating materials three times.

The sludge-drying experiment was carried out using the apparatus shown in Fig. 2. The water content was calculated as below.

$$\text{Initial water content (\%)} = (S_1 - S_3) / S_1 * 1$$

$$\text{Water content after drying (\%)} = (S_2 - S_3) / S_2 * 100$$

S_1: weight of Sludge (g)
S_2: weight of Sludge after microwave drying (g)
S_3: Weight of fully dried sludge (g)

2.1.4. Characterization

The X-ray diffraction (XRD) pattern of the heating materials to find crystal growth and structure was obtained by an Empyrean, (Malvern Panalytical, UK) employing a Cu Ka radiation source operating at 40 kV and 40 mA. Data were collected over the 2 theta degree, range of 5–90° using a scan rate of 0.0333°.

Table 2
Notation of the prepared heating materials.

Notation	Main material	Additive	Weight
A	$LiNO_3$:$6Fe_2O_3$: ZnO	–	–
B	$LiNO_3$:$6Fe_2O_3$: ZnO	SiC	10 wt%
C	$LiNO_3$:$6Fe_2O_3$: ZnO	carbon powder	3 wt%
D	$LiNO_3$:$6Fe_2O_3$: ZnO	carbon nano fiber (CNF)	10 wt%
E	$LiNO_3$:$6Fe_2O_3$: ZnO	Nickel nitrate hexahydrate (precursor)	10 wt%
F-1	$LiNO_3$:$6Fe_2O_3$: ZnO	Nickel powder	5 wt%
F-2	$LiNO_3$:$6Fe_2O_3$: ZnO	Nickel powder	10 wt%
F-3	$LiNO_3$:$6Fe_2O_3$: ZnO	Nickel powder	15 wt%
F-4	$LiNO_3$:$6Fe_2O_3$: ZnO	Nickel powder	30 wt%
F-5	$LiNO_3$:$6Fe_2O_3$: ZnO	Nickel powder	50 wt%
F-6	None	Nickel powder	100 wt%
G-1	$LiNO_3$:$6Fe_2O_3$	–	–
G-2	$6Fe_2O_3$:ZnO	–	–
G-3	$LiNO_3$:$6Fe_2O_3$	Nickel powder	10 wt%
G-4	$6Fe_2O_3$:ZnO	Nickel powder	10 wt%

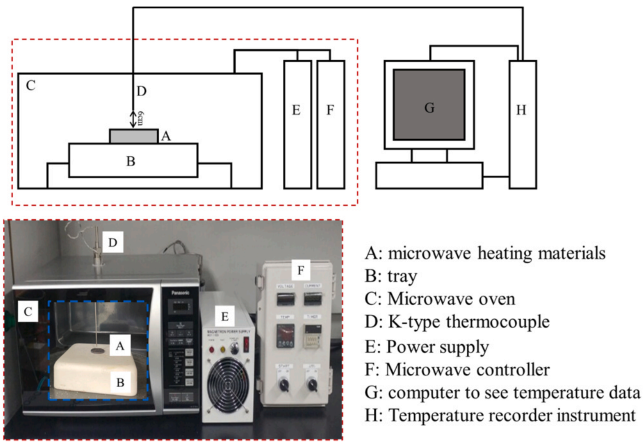

Fig. 1. Schematic diagram of microwave heating process.

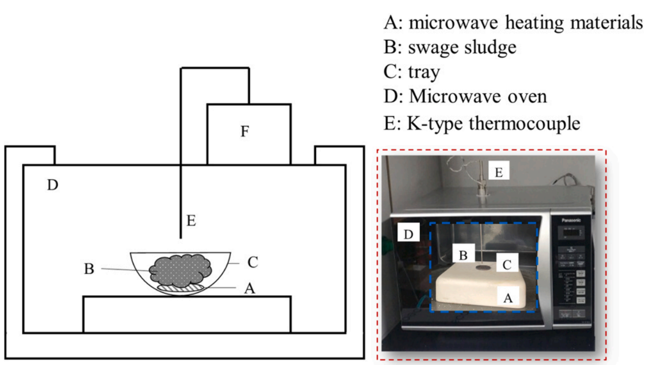

Fig. 2. Schematic diagram of sludge drying process.

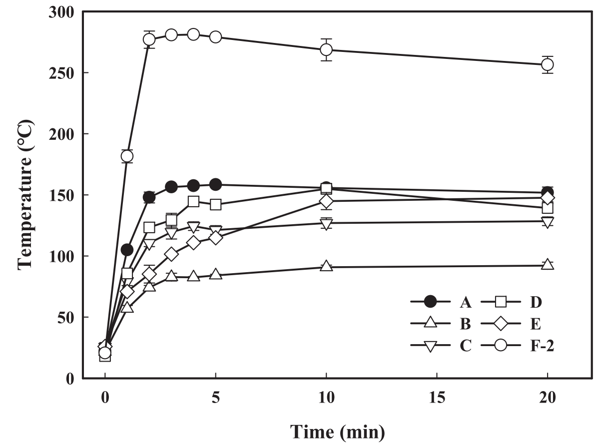

Fig. 3. Exothermic performance (500 W, 20 min) of heating materials with A, B, C, D, E and F-2.

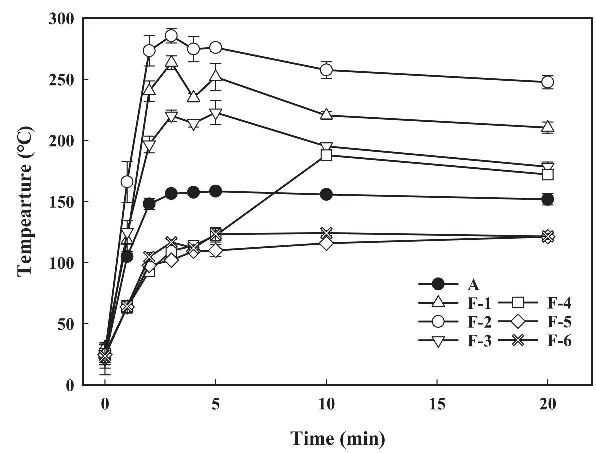

Fig. 4. Exothermic performance of heating material with different nickel wt%. (Microwave injection: 500 W, 20 min; heat treatment: 1400 °C).

3. Results and discussion

3.1. Effect of manufacturing process in heating performance

3.1.1. Evaluation of the heat generated by materials with various additives

In general, microwave penetration depth is highly dependent on the composition of microwave radiated materials, so the penetration depth of a microwave in heating material fabricated in this study is several tens of times deeper than that of ceramic based materials. However, if the material is too thin than for the microwave penetration depth, only a partial microwave energy is absorbed and the rest is reflected. To more efficiently utilize the energy, metallic materials such as Fe and Al are mixed for the unabsorbed energy to transfer to the metal ions and to supply energy to the ceramic material again [22]. As mentioned above, a study by Chandrasekaran on the carbon-source graphite and Fe-source magnetite showed that a strong microwave absorbing material such as copper, manganese and SiC was can sustain high temperatures and provide high heating rates. However, they generate less heat when magnetite was converted into Fe or FeO under microwave excitation [29]. Jang et al. also reported that when $Li_5Fe_5O_8$ was formed on the surface of heating materials, Fe_2O_3 was not converted to Fe or FeO even after microwave exposure [28]. Therefore, the enhancement of microwave sensitivity can be achieved when the additive was partially mixed with the Li-Fe-Zn mixed material to induce the formation of Li-Fe compounds, for improving the heating performance (Figs. 3 and 4).

It was shown that the sample F-2 exhibited highest thermal response, as shown in the Fig. 3. The thermal response of A, B, C, D, E and F-2 was 155.8 °C, 90.8 °C, 126.9 °C, 155 °C, 145 °C and 269 °C, respectively. Except for the F-2, the performance of samples were lower than that of A without any additives. Nickel was known to form $NiFe_2O_4$ species by interacting with Fe_2O_3 or Fe_3O_4 and to have exothermic performance in the magnetic field [30,31]. Especially, when SiC was added to the sample B, no exothermic performance was observed in the microwave field because the agglomeration of SiC generated SiO_2 species. Zhou et al. reported that coating of iron particles with SiO2 reduced the current loss and ferromagnetic resonance of iron particles. It was shown that SiO_2 interferes with iron particles which was the main exothermic component in the study [32]. A similar study by Liu et al. also reported that SiO_2 coated Ti_3SiC_2 material improved microwave absorption [33]. Wang et al. reported that SiO_2 coated carbonyl iron/polyimide material reduced the minimum reflection loss from 25 dB at 1.7 mm. It means that the SiO_2 increased the absorption rate of microwaves and have reduced the reflection loss, thereby reducing the exothermic performance [34]. As indicated by a number of previous works, formation of SiO_2 acts as and impediment for the Li-Fe-Zn heating material. Carbon based C and D also showed little exothermic performance in the microwave field. It was a reason that CNF (Carbon nano fiber) was a fiber made of carbon, which prevents exothermic due to the low dielectric constant of the fiber itself.

3.1.2. Evaluation of the heating performance of the heating material with different additive content

It was found that the heating performance was enhanced when nickel powder was added. To find the optimal amount of nickel powder to be added, 0–100 wt% of the powder was added to the mixture. Fig. 4 shows the heating performance by various contents of the materials prepared.

As a result, the F-2 exhibited the best exothermic performance at 182 °C for 1 min and 269 °C for 10 min, followed by the sample F-1 at 118 °C for 1 min and 220.5 °C for 10 min. F-6, which contains the highest amount of nickel, showed no performance improvement and the maximum exothermic temperature was 124 °C. These results indicate that the metallic nickel species does not heat up, however, if mixed as a small amount of additives, the nickel added compound can generate heat much more efficiently. Similarly, Lysenko reported that a compound such as Ni-Li-Zn-Ferrite could be used as a microwave-absorbing material [35]. In addition, Yuan reported that adding a nickel to SiC could broaden the microwave absorption bandwidth [25]. To verify the existence of Fe, XRD analysis was performed(Fig. 5). Among all the heating materials containing Nickel, NiO species with peaks at 37.2°, 43.2°, and 62.8° were observed, and the NiO peak increased as the contents increased. It is believed that the NiO species also became dominant because of the presence of excess nickel on the surface of the sample. When excessive nickel was present, F-6, it was predominantly produced by compression containing only nickel and lignin, which is a binder, showed a dominant distribution of NiO. Therefore, it was showed that as the amount of Ni added increased, NiO species became dominant and no other species could be formed. It is worth noting that XRD analysis of the F-2 confirmed $NiFe_2O_4$ species on the surface of the heating block and no binding of the Zn. Also, the heating performance was improved without generation of Zn compounds. The $NiFe_2O_4$ species were believed to possess a good absorption properties and thus serve various applications in the microwave absorption fields [36].

In this study, Metal oxides such as lithium, ferric oxide and zinc reported in various literatures were mixed. But a result of the XRD analysis, the peak of Ni-Li-Fe-O composite oxide was observed and there was no peaks from zinc compound. Therefore, the heating performance was showed in Fig. 6 when the ternary material was divided and mixed into the binary material.

The temperature of G-1 without zinc oxide was 171.5 °C, and G-2 without lithium nitrate was 113.5 °C. Through the results of performance, lithium served as a substance that enhances the heating performance in the microwave field while zinc didn't show a significant effect. In addition, nickel added G-3 and G-4 reached 230.7 °C and 143 °C, respectively. It was concluded that the addition of nickel can improve the heating performance in all materials tested in this study, and that the addition of lithium was essential for improving the heating performance.

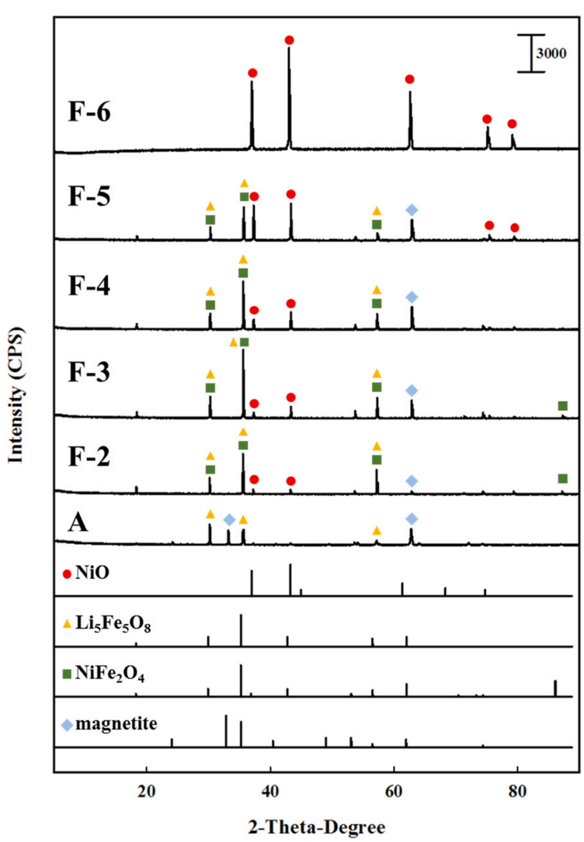

Fig. 5. XRD patterns of different nickel wt%.

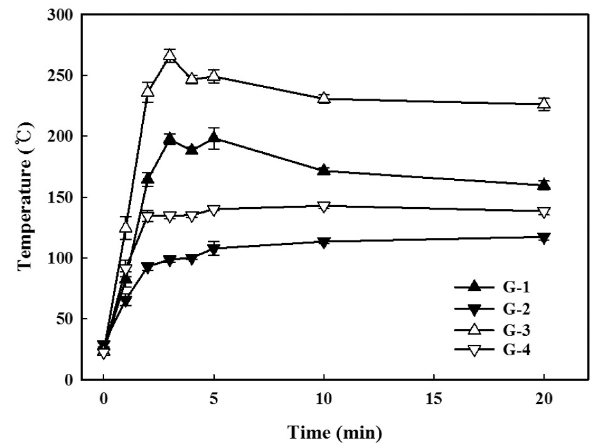

Fig. 6. Exothermic performance of heating material on the binary material. (Microwave injection: 500 W, 20 min; heat treatment: 1400 °C).

3.2. Stability and durability of heating material

3.2.1. Evaluation of heating material's reproducibility and stability

A heating material that generates heat in response to microwaves can form a dry atmosphere by raising the ambient temperature of the drying chamber due to the increase of the drying efficiency. Among samples tested in this study, F-2 was found to be the most suitable material for forming the dry atmosphere. In addition, in order to confirm the stable heating performance during prolonged period for microwave drying, it was necessary to perform cyclic heating and cooling. Fig. 7 shows the performance after multiple microwave exposure to verify microwave stability of the heating block. After cyclic heating experiment, XRD analysis was conducted to verify the crystalline status of the material formed on the surface (Fig. 8).

It was showed that even when the material was irradiated 10 times, the heating performance was maintained with no signs of deterioration. As the number of irradiations increased, the species on the surface of

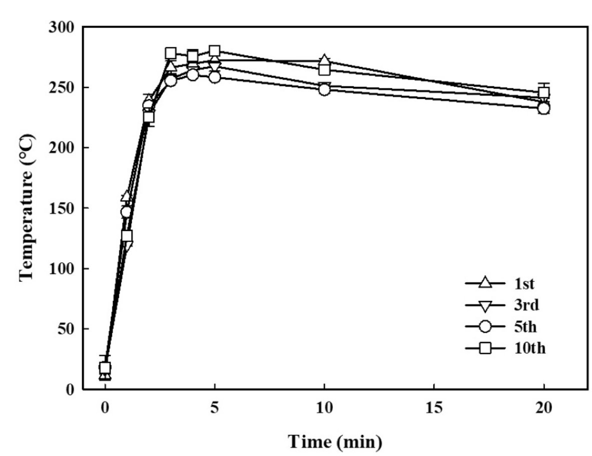

Fig. 7. Exothermic performance of microwave irradiation frequency. (Microwave injection: 500 W, 20 min; heat treatment: 1400 °C).

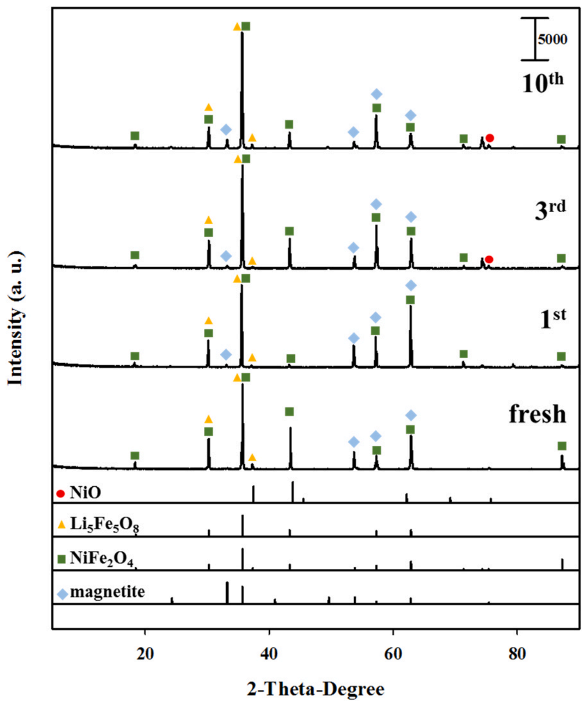

Fig. 8. XRD patterns of different microwave irradiation frequencies.

materials didn't change, and each species were observed at the following degrees. $Li_5Fe_5O_8$ was found at 37.2, 43.2 and 63.7, $NiFe_2O_4$ was found at 30.62, 36.05, 37.72, 43.72, 54.12, 54.72, 63.32 and 87.2, and Fe_2O_3 was observed at 33.22, 35.54, 35.8, 43.2, 53.8, 58, 62.88. In addition, Exposing Fe_2O_3 or Fe_3O_4 to microwave allows high exotherm, but was reduced to FeO to drastically reduce performance. However, even after repeated irradiation when present as the metal oxide mixtures, it remained surface species such as $Li_5Fe_5O_8$, $NiFe_2O_4$, etc. to ensure optimal heating performance and stability. As a result, even when repeatedly exposed to microwaves, the heat generation performance was not weakened, and the optimum heat generation element F-2 was maintained.

3.2.2. *Characterization of sludge drying process by heating materials*

These technologies can reduce energy consumption and improve energy efficiency, thereby reducing greenhouse gas emissions. Table 3 shows the sludge moisture content with or without heating material in microwave drying facilities. It was shown that when the drying system using F-2 was dried for about 5 min, high quality sludge having a moisture content of less than 1% was discharged. It shows the drying efficiency was improved by over 43% compared to microwave drying without heating material. Therefore, the drying time of the microwave drying system can be further shortened and the high quality dry sludge emissions per hour can be increased. As a result, it has been found that heating materials can increase efficiency during microwave drying.

4. Conclusions

To increase the energy efficiency of the drying process, the heating performance and the sludge drying performance of various additives were evaluated. In addition, XRD analysis was performed to confirm the surface structure of heating materials. When F-2 heating material was heat treated at 1400 °C in a N_2 atmosphere, the maximum performance reached at 269 °C. The XRD result showed the $Li_5Fe_5O_8$ species, reported as the heating species, was formed. Also, the $NiFe_2O_4$ species was found to be dominant on the surface. In contrast, it was found that as the content of added nickel increased, more NiO species were formed than $Li_5Fe_5O_8$ and $NiFe_2O_4$ species and the heating performance also

Table 3
Moisture contents and drying efficiency with material or without.

Time (min)	Moisture contents (%)		Drying efficiency (%)
	Without	With F-2	
0	34.5	34.5	
1	27.0000	21.8610	43.3025
5	5.3676	0.5006	21.7829
10	3.0273	0.2906	8.7747

decreased. Therefore, as a result of drying the sludge by applying the optimal heating material, the drying efficiency was increased by 43% and obtained the dry sludge having a moisture content of less than 1%. Thus, the applicability of microwave heating materials has been demonstrated as a way to maximize the energy efficiency of the drying process to facilitate greenhouse gas reduction and save energy.

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CRediT authorship contribution statement

Younghee Jang: Conceptualization, Methodology, Writing - original draft. **Hanki Eom:** Formal analysis, Investigation. **Soon Woong Chang:** Formal analysis, Validation. **Sang Moon Lee:** Supervision, Project administration. **Sung Su Kim:** Supervision, Project administration.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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