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Development of Density Control Technology for Improving Medical Radiation Shielding Performance of Waste Marble Powder Mixture

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Abstract: The marble used at construction sites creates a large amount of sludge after processing. Because waste marble has a high calcium oxide content, it is often used as a concrete mix building material. In this study, the use of waste sludge in the fabrication of radiation shields was investigated for medical shielding applications. A shielding sheet was produced by mixing a polymer and waste marble powder. A method for improving the density of the shielding sheet was developed to improve the shielding performance. To improve the density of the shielding sheet using the WMP mixture, the gap between particles was narrowed by making the WMP particles small and by mixing in a material with a proven shielding effect, such as bismuth oxide. In addition, a stirring defoaming process was used to reduce the voids between particles, and we presented a method to control the density by processing the WMP at a high temperature of 1200 °C. The experimental results revealed that the waste marble powder exhibited the highest shielding effect when mixed with radiation shielding materials such as bismuth oxide. The reduction of voids and the size of the particles used in preparing the shielding sheet proved to be effective in reducing the gap between the particles, resulting in an improvement of shielding of approximately 15% to 20%. The investigated shielding material based on waste marble powder was shown to be effective in shielding low-dose radiation.

Keywords: medical radiation; waste marble; plastic working; agitation defoaming; bismuth oxide

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Citation: Kim, S.-C. Development of Density Control Technology for Improving Medical Radiation Shielding Performance of Waste Marble Powder Mixture. *Coatings* **2023**, *13*, 272. https://doi.org/10.3390/coatings13020272

Academic Editor: Valentin Craciun

Received: 3 January 2023
Revised: 19 January 2023
Accepted: 20 January 2023
Published: 25 January 2023

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1. Introduction

Marble is the result of the metamorphosis of sedimentary rocks such as carbonate-based limestone or dolomite. After the recrystallization of carbonate minerals, a marble structure is created in the form of interlocking crystals [1,2]. The non-white hue is caused by impurities, such as mud, silt, sand, and iron oxide [3]. Marble is mainly used as a building and interior decoration material; therefore, it is typically processed into a desired shape. The waste generated during marble processing is produced in the form of sludge [4]. When the sludge is dried, it is converted into a powdered form, which is generally difficult to process and manage, and adversely affects the environment [5]. Waste marble powder (WMP) is often recycled as cement, a building material, because of its high calcium oxide content; and affects the strength and modulus of elasticity of composites [6].

This study focused on fabricating a radiation shield using waste materials that are inferior in shielding performance to those of conventionally used shielding materials such as tungsten or lead, but need removing from the environment due to their negative impact [7]. Shields based on WMP were fabricated in the form of fibers or sheets to facilitate flexibility; however, it is necessary to reduce gaps and voids between particles during the manufacturing process [8].

The primary objective of this study is to develop a material that is suitable for shielding applications in medical institutions by utilizing a composite material consisting of WMP and polymers. To recycle marble waste as a radiation-shielding material, it must be determined to be suitable as a composite material. Lead, which is mainly used in the

production of shielding sheets, has a high atomic number and density, and is excellent from the perspectives of cost and processability. In addition, because of its excellent affinity to polymer materials and ability to induce uniform dispersion inside a shielding sheet, the reproducibility of shielding performance is high even when used as a single shielding material [9]. WMP has a high calcium oxide content (CaO, density 3.34 g/cm^3), in addition to high ionization and porosity. Therefore, it is difficult to mix it with other materials [10]. Given that the purpose of a shielding sheet is to achieve excellent radiation shielding performance, WMP, which is a type of industrial waste, cannot be used without being processed. Thus, the suitability of the shielding material can only be discussed when the WMP is processed or mixed to obtain new characteristics. In previous studies, shielding sheets were prepared in the form of fibers impregnated with yarn; however, as time elapsed, peeling due to hardening occurred [11].

To address this problem, a process technology for improving the performance of prepared shielding sheets using WMP is presented. In particular, a method for ball milling coarse-grained and large-sized WMP particles and an approach for preparing composite materials by incorporating bismuth oxide, an eco-friendly material with excellent economic efficiency and processability, are proposed. In addition, this study aims to improve the shielding performance by increasing the density of the shielding sheet using an agitation defoaming process for liquid mixtures that reduces gaps and voids between particles when mixed with polymers, and plastic processing that increases the affinity of the WMP particles and the polymers.

In particular, the medical radiation-shielding material using WMP has an economic advantage over other materials and, when used as a composite material, the advantage of being used as a lightweight material.

2. Materials and Methods

To reduce the intensity of the incident radiation, the thickness of the shielding sheet must be increased to increase the probability of absorption during penetration [12]. This implies an increase in the density of the atoms or molecules that interact with the incident radiation inside the shielding sheet, rather than simply increasing the thickness. Therefore, the density of a WMP sheet refers to the interaction between the mass per unit area and the incident energy for a specific medium [13]. The interaction of incident radiation can be calculated using the Beer–Lambert law [14]:

$$I = I_0 e^{-\mu t}, \tag{1}$$

$$\mu_m = \left(\frac{\mu}{\rho}\right) = \left(\frac{In\left(\frac{I_0}{I}\right)}{\rho t}\right) = \frac{In\left(\frac{I_0}{I}\right)}{t_m}, \tag{2}$$

where I_0 and I are the incident and transmitted photon energy intensities, respectively; μ (cm^{-1}) and μ_m (cm^2/g) are the linear and mass attenuation coefficients, respectively; t (cm) and t_m (g/cm^2) are the thickness of the shielding sheet and the thickness per unit mass, respectively; and ρ (g/cm^3) is the material density.

Therefore, to increase the density per unit area of the shielding sheet, more WMP must be added. This requires a reduction of the spacing between particles; i.e., the voids in the material. When a polymer material and WMP are mixed, pores of various sizes are generated [15]. Therefore, the size of the particles must be reduced to minimize the size of the pores. The higher the density, the smaller the porosity, the smaller the particle size, and the smaller the space in which the pores can be distributed. Therefore, the total porosity (P_s) of the shielding sheet can be calculated using Equation (3) using the density (D_P) of the mixture of the polymeric material and the WMP, and the density (D_{WMP}) of the WMP [16]:

$$P_S = 1 - \frac{D_{WMP}}{D_P}, \tag{3}$$

This study investigated the increase in the density of the shielding sheet using ball milling to process the particles of the waste marble powder into small particles and defoaming to remove air from the mixture.

First, shielding sheets with an average thickness of 1 mm were manufactured using the same amount of WMP to evaluate the shielding performance. High-density polyethylene (HDPE) was used as a mixed polymer material. HDPE has excellent strength and is primarily used as a disposable plastic product [17]. The PE had a molecular weight of $\geq$ 4 million and a density of 0.91 g/cm^3. To improve the shielding performance, a sheet incorporated with bismuth oxide was produced, and its mixability with other materials was examined [18].

In the process of manufacturing the shielding sheet, pretreatment was performed to reduce the voids and improve the hydrophilicity of the polymer material; thereby, increasing the dispersibility of the WMP. To reduce the particle size, ball milling (ball milling machine, Henan Mining Machining Manufactory, Zhengzhou, China) was performed twice for 3 h, as shown in Figure 1. The particle dispersion inside the WMP shielding sheet was visually compared and reviewed before and after ball milling.

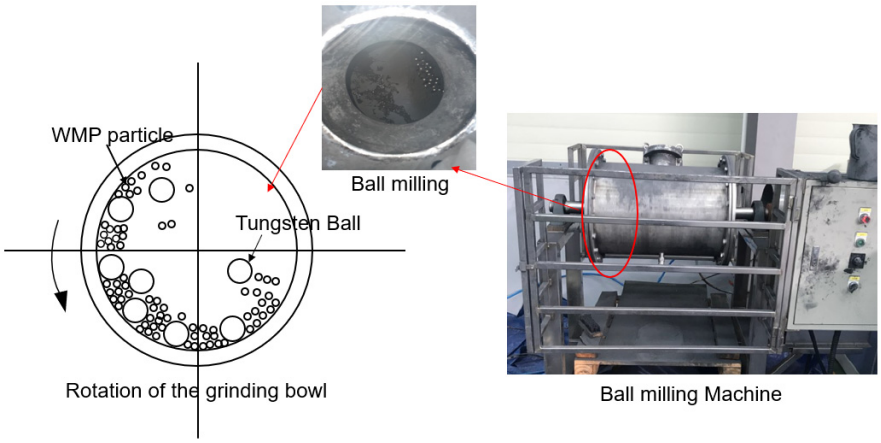


Figure 1. Ball milling process using tungsten ball.

To reduce the air gaps generated by the bubbles inside the sheet during the manufacturing of the shielding sheet, agitation defoaming was performed using an agitation defoaming machine (agitation defoam machine, HS 2.0, Daegu, Korea) [19] during mixing with the liquid polymer material. The shielding performance was compared and analyzed based on the density changes before and after the defoaming.

To produce the WMP sheet, the powder generated after marble processing was dried at 80 °C for 6 h before use. After adding WMP to a liquid polymer, the particles were dispersed by stirring at 5000 rpm. To achieve uniform shielding performance of the casting solution, impurities were removed via filtering, followed by a defoaming process. The final step was the lamination process of compression molding. The resulting shielding sheet was 100 mm $\times$ 100 mm $\times$ 1 mm.

The size of the particles and dispersibility of the shielding material were observed with an optical microscope (FESEM; field emissions scanning electron microscope, Hitachi, S-4800, Korea), using thin film slices of the shielding sheet [20]. To evaluate the shielding performance, 10 experiments were performed using an X-ray generator (Toshiba E7239, 150 kV–500mA, 1999, Tochigi-ken, Japan), and the average value was recorded. The medical radiation used in the experiments was converted into effective energy, which was a single energy source. The half-value layer (HVL) for converting the medical radiation used in the experiment into effective energy, which is a single energy source, can be measured by taking the log_e of both sides in the Beer–Lambert law ($I = I_0 e^{-\mu x}$) and calculating the slope from the graph of $y = -ax$. After obtaining the value for the linear attenuation coefficient μ from this slope, it was determined that HVL = 0.693/μ [21]. In addition, Hubbell's

mass attenuation coefficient table was used to calculate the effective energy, which has the same value as the HVL corresponding to the single energy source of the HVL [22]. The tube voltage used in the X-ray generator was 40–120 kVp, 30 mA. The dose detector was calibrated using an ionizing dosimeter (TnT 12000; FLUKE Corp., USA). The absolute value of the radiation shielding standard was based on a 0.1 mm lead plate.

To evaluate the shielding performance, a radiation experiment was performed, as shown in Figure 2; and the shielding rate (*S*) of the manufactured shielding sheet was calculated using Equation (4). *W* is the measured radiation dose when a shielding sheet is placed between the X-ray beam and detector, and W_0 is the measured radiation dose without a shielding sheet between the X-ray beam and detector [23]:

$$S = \left(1 - \frac{W}{W_0}\right) \times 100\%, \quad (4)$$

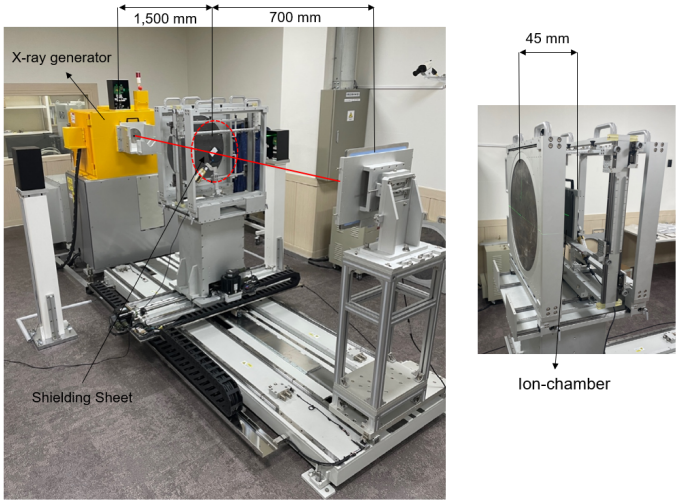


Figure 2. Evaluation of radiation shielding performance of shielding sheets.

Using this process, changes in the particle size, arrangement, and shielding rate of the shielding sheet were observed.

3. Results

To manufacture an X-ray shielding sheet using WMP for use in medical institutions, samples were prepared under five different conditions: (1) processing marble powder collected from a business site; (2) ball milling the collected WMP to an average particle size of 4 μm; (3) mixing WMP with bismuth oxide (15 wt.%) and stirring; (4) reducing internal voids through a defoaming process; and (5) mixing WMP with a polymer before plastic processing at 1200 °C. Table 1 shows the composition characteristics of the manufactured sheets.

Table 1. Properties of the WMP shielding sheets for different processing conditions.

Item	Value				
	WMP	**WMP (Ball Milling)**	**WMP (+Bi_2O_3)**	**WMP (Defoaming)**	**WMP (Plastic Work)**
Sheet structure	Single structure				
Mixing ratio (polymer to WMP)	1:3				
Sheet weight (kg/m^2)	2.50	2.15	3.15	2.18	2.12
Sheet thickness (mm)	1.01	0.92	1.43	0.89	0.75
Polyethylene (g)	83.4	83.5	84.0	82.4	80.5
WMP (g)	250				

The manufactured shielding sheets are illustrated in Figure 3. Sheets were made using the same amount of WMP, and the difference in thickness was the result of the processing and mixing. The sheets were manufactured using a lamination process, and changes in the internal density were observed.

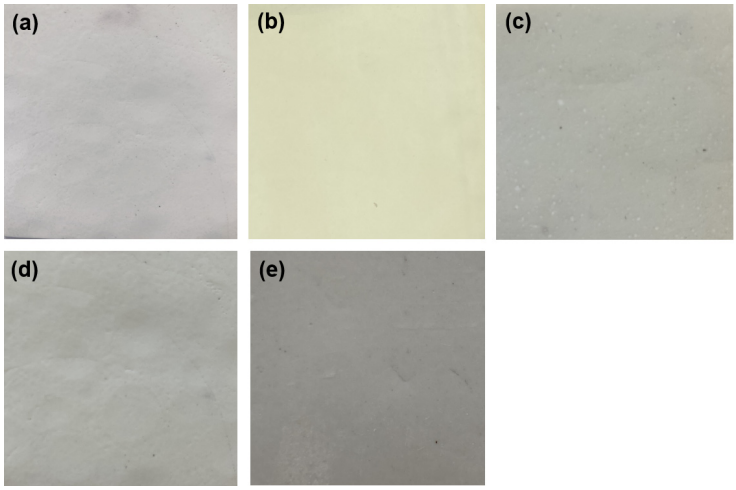


Figure 3. Appearance of the shielding sheet produced for the shielding performance comparison test: (**a**) single sheet of waste marble powder; (**b**) sheet made from a mixture of waste marble powder and bismuth oxide; (**c**) sheet made of waste marble powder after ball milling; (**d**) defoaming sheet made by removing air from the waste marble sheet; and (**e**) sheet produced after plastic processing (1200 °C) of waste marble powder.

The internal cross sections of the final fabricated sheets were visually compared and analyzed. Figure 4 compares the cross sections of a sheet manufactured via ball milling WMP and a sheet without ball milling. It can be seen that the rough surfaces of the particles disappear slightly.

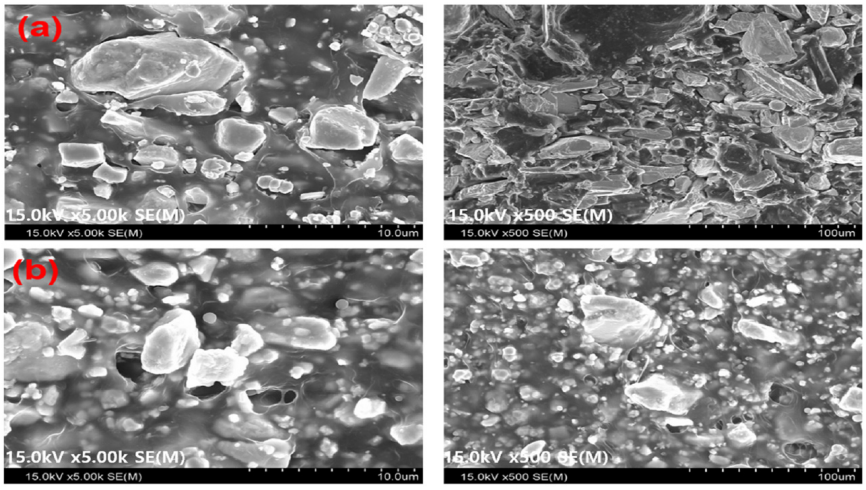


Figure 4. Cross-sectional comparison of WMP shielding sheets: (**a**) cross section of a WMP shielding sheet; (**b**) cross section of a WMP shielding sheet processed using ball milling.

Figure 5 shows an enlarged cross section of the shielding sheet mixed with bismuth oxide. Figure (a) confirms that the aggregation between bismuth oxide particles and MWP particles occurred because of the high affinity between the particles. In addition, it can be predicted that the overall density can be improved by reducing the number of voids.

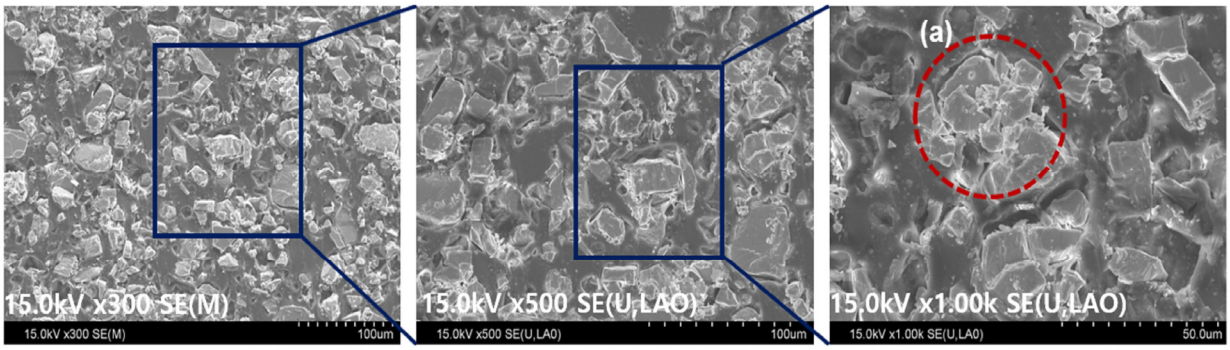


Figure 5. Cross-sectional view of a shielding sheet mixed with WMP and bismuth oxide.

Figure 6 compares the internal cross section of the sheets based on the defoaming process. It can be observed that many bubbles are generated, as in Figure 6a, in the mixing process with the polymer material.

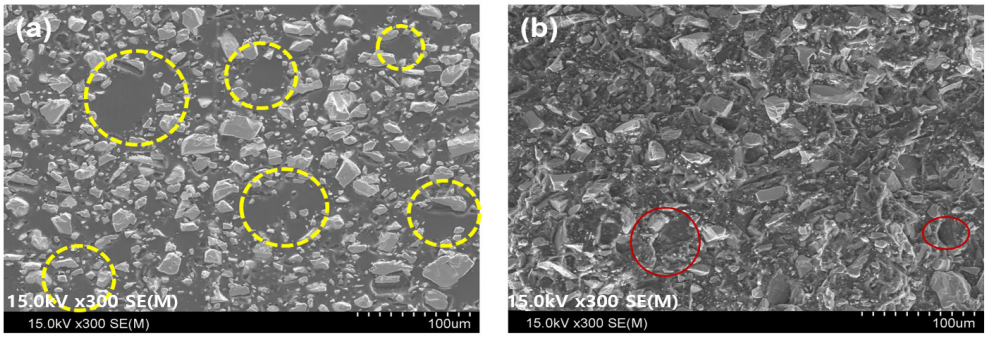


Figure 6. Comparison of cross sections of shielding sheets based on defoaming using an agitation defoaming machine in the WMP fabrication process: (**a**) is a sheet without defoaming; (**b**) is a sheet with defoaming.

Figure 7 is a cross-sectional comparison of the sheets manufactured by plastic processing the sludge-based WMP generated during waste marble processing. Figure 7b confirms that the size of the particles is small, the roughness of the particles improved, and the distribution of the polymer material is reduced.

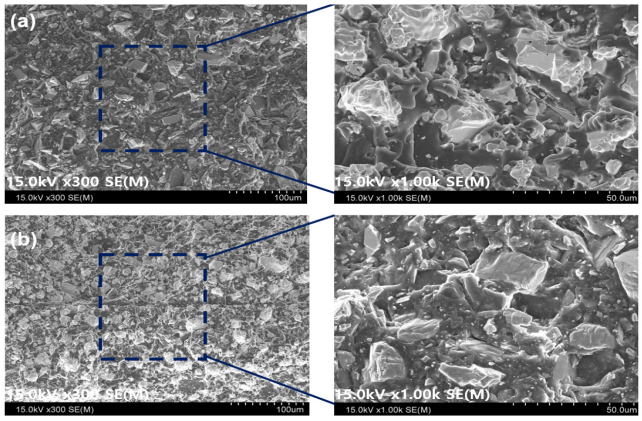


Figure 7. A cross-sectional comparison of the WMP shielding sheets produced using plastic working: (**a**) a sheet without plastic processing; (**b**) a sheet subjected to plastic processing at 1200 °C.

The shielding performances of the final fabricated sheets were compared. The shielding rates of the shielding sheets manufactured using the aforementioned processes are

shown in Table 2. A comparison of the results reveals that the sheet containing bismuth oxide was the best, and the shielding ratio of the sheet subjected to ball milling or defoaming was higher than that of the sheet manufactured without WMP processing. In particular, in the case of the plastic-processed sheet, the shielding rate was significantly higher; in addition, it was confirmed that the effect of reducing the spacing between the particles led to improved shielding performance. Therefore, plastic working was the most effective method to maximize the shielding effect by narrowing the gap between the particles of WMP, and this method showed the effect of increasing the shielding rate in both low- and high-energy regions. When bismuth oxide and WMP were mixed, the shielding rate increased by approximately 193%. Compared to lead, which is the absolute value of shielding performance evaluation, it shows a very low shielding rate. Therefore, it can be seen that there are limitations as an independent shielding material of WMP.

Table 2. Comparison of shielding performance of sheets manufactured using different processing approaches.

Radiation Type	Effective Energy (keV)	Shielding Rate (%)					
		Lead (0.1 mm)	WMP	WMP (Ball Milling)	WMP (Defoaming)	WMP (Plastic Work)	WMP (+Bi_2O_3)
X-ray	23.5	81.54	31.2	36.4	39.4	58.9	72.2
	27.4	83.47	26.2	27.4	34.2	56.4	68.8
	33.1	87.54	20.5	22.5	30.0	41.5	60.4
	47.9	90.25	18.4	19.1	24.9	35.2	54.1
	55.4	97.04	12.1	12.5	23.4	30.1	52.4

4. Discussion

Radiation shielding in medical institutions can be divided into direct and indirect shielding [24]. One of the most important factors is the effect on the physical activity of medical personnel. If the shield is made of thick and heavy material, this may limit the wearer's mobility during medical practice. Therefore, it is necessary to develop a variety of materials to achieve the appropriate shielding effect for each energy level of radiation exposure [25]. Although there are many alternatives to lead, the development of shields using discarded materials, such as waste plastics, waste shells, and waste marble, is desirable in terms of environmental protection [26,27]. However, cost and shielding performance are critically important factors. Shield production using waste marble is different from the existing waste marble. In the case of existing waste, cost and time are input through the reprocessing process; however, in the case of WMP, since it is processed and reproduced in the form of sludge, it can be mixed with polymer immediately after removing impurities; thus, there is an economic advantage.

Therefore, in this study, a shielding sheet was manufactured using waste marble sludge. The technology used in the manufacturing process increased the density of the material by reducing the gap between the constituent particles. If the density is increased, the probability that an incident radiation photon will interact with the cross-sectional area increases, which can improve the shielding performance [28]. The strategies used to improve shielding performance included reducing the particle size via ball milling, reducing internal voids via defoaming, increasing the affinity between WMP particles by plastic working at a high temperature, and the use of other shielding materials for further mixing. To effectively increase the shielding performance, increasing the amount of shielding material proved to be the most effective. However, effective absorption and scattering should occur inside the shielding sheet as much as possible, considering the weight [29–31].

The results revealed that the increase in the shielding performance achieved by mixing other materials, for which the shielding effect was already verified, was the highest. Other methods used to increase the density also improved the shielding effect by reducing the gap between the shielding particles in the polymer material.

Waste marble is used in the construction of concrete buildings and in the preparation of shielding fibers [32–35]. Although the shielding suit used in medical institutions consists of fibers, it is difficult to verify the reproducibility of the shielding effect owing to the

pinholes associated with the weaving process of the yarn material, and the shielding effect is inferior owing to the thickness [36]. Therefore, shielding suits are primarily made of film and sheet materials, wherein appropriate flexibility can be achieved by mixing shielding and polymer materials in the manufacturing process [37].

In this study, marble powder, which is recognized as construction material, was fabricated into thin sheets for use in radiation shielding. Although the sheets exhibited a lower shielding rate than tungsten, which can replace lead, it is a promising radiation shielding material; in addition, enhanced shielding may be possible using different strategies, such as adjusting the mixing ratio and solid polymer mixing. The suits used in medical institutions should be made of eco-friendly materials, and efforts should be made to develop new materials that satisfy this requirement [38–40].

These radiation shielding materials can be used under various conditions in the future. If shielding using the energy domain is required, finding economical materials will be the most important condition; hence, mixture research in the field of recycling will be important, as in this study. A limitation of this study is that the shielding effect could not be verified when compared to the mixing mass of the WMP and high-density polyethylene; therefore, not considering the shielding effect according to the wt.% of WMP. This is because the higher the wt.%, the higher the shielding effect. For shielding in the field of medical radiation, it is necessary to develop new materials that minimize weight and thickness.

5. Conclusions

A fabricated sheet capable of shielding medical radiation was produced using WMP, sludge generated after marble processing at an industrial site. Four process technologies were investigated to improve the shielding performance. The sheet with the best shielding effect was made of a material mixed with other shielding materials. The next most effective process technology reduced the interparticle spacing via plastic working of the WMP, and the manufactured sheet exhibited improved shielding by 91.3% or more in the high-energy region. The ball milling and defoaming processes used to reduce the air gap also increased the shielding effect by approximately 5% to 10% or more. Therefore, WMP can be used to manufacture radiation shielding sheets in medical applications.

Funding: This work was supported by the National Research Foundation of Korea (NRF) grant funded by the Korean government (MEST) (NRF. 2020R1I1A3070451). This work was supported by Radiation Technology R&D program through the National Research Foundation of Korea funded by the Ministry of Science and ICT (2020M2C8A1056950).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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