

Feasibility Study of the Paper Sludge Properties in Manufacturing Recycled Filler

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The mineralization of inorganic components in paper sludge offers potential for their re-use in various fields. This study synthesized carbonates based on the minerals present in paper sludge and aimed to investigate the control of particle size distribution and particle shape of the synthesized carbonates. First, the physicochemical and thermal properties of paper mill sludge and ash were evaluated using a spectrophotometer, XRF, and TGA. Paper sludge ash was obtained by incinerating the paper sludge, followed by a carbonation reaction using CO₂ gas injection and pH neutralization to produce recycled fillers. The average particle size, crystal lattice structure, and morphology of the recycled fillers were analyzed using a particle size analyzer, XRD, and FE-SEM. The results indicate that the recycled filler produced from coating paper sludge exhibited similar brightness and morphology to conventional paper-based fillers, suggesting its potential to replace commercial PCC (Precipitated Calcium Carbonate). In contrast, the recycled filler manufactured using old corrugated container mill sludge was deemed unsuitable due to its low hydration activity.

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INTRODUCTION

Paper sludge can be defined as the final solid waste resulting from the process of wastewater treatment at a paper mill. Although paper sludge is released *via* physical and biological processes, it is recognized as a substance that can cause environmental problems because of its high volume. Under the ratification of the London Convention in 1972, marine discharges and off-site incineration of wastes including paper sludge were regulated (Convention 1996). In 2016, the Ministry of Environment of the Republic of Korea announced that the wastes treated by marine discharges were to be transferred to full-scale land treatment processes (Ministry of Environment 2008). Therefore, domestic paper companies began constructing new facilities and introducing technologies to reduce marine discharges. Consequently, the production of domestic paper sludge has been decreasing on a year-on-year basis since 2013 to 9.2% in 2017 (as shown in Fig. 1). However, according to the Korea Paper Association, around 1,273,000 tons of paper sludge were still generated in 2017. Therefore, alternative eco-friendly treatment technologies should be investigated.

Paper sludge specimens from different sources are mainly composed of moisture, short fibers, and inorganic components. In terms of short fibers and inorganic components, their constituent characteristics offer the possibility of their re-use in various areas. For instance, as short fibers consist largely of cellulose, Taramian *et al.* (2007) applied recovered fibers from sludge to the manufacturing process of particle boards. Short fibers

can also be used as a biomass energy source. Gavrilescu (2008) conducted a study that used paper sludge as a fuel, while Fan and Lynd (2007) and Gavrilescu (2008) investigated the production of ethanol by treating cellulase in paper sludge. The mineralization of inorganic components was studied using ash, a by-product from the heat treatment of paper sludge. Wajima and Munakata (2012) synthesized K-F zeolite as a land modifier, while García *et al.* (2008) prepared absorbents for the removal of oils and used chemicals. Both Likon *et al.* (2011) and Liaw *et al.* (1998) studied the use of paper-material sludge as a building material and cement admixture in light of its pozzolanic properties. Kim and Kim (2018) and Lee *et al.* (2015) used paper sludge ash as a greenhouse-gas storage medium using carbon capture, utilization, and storage technology.

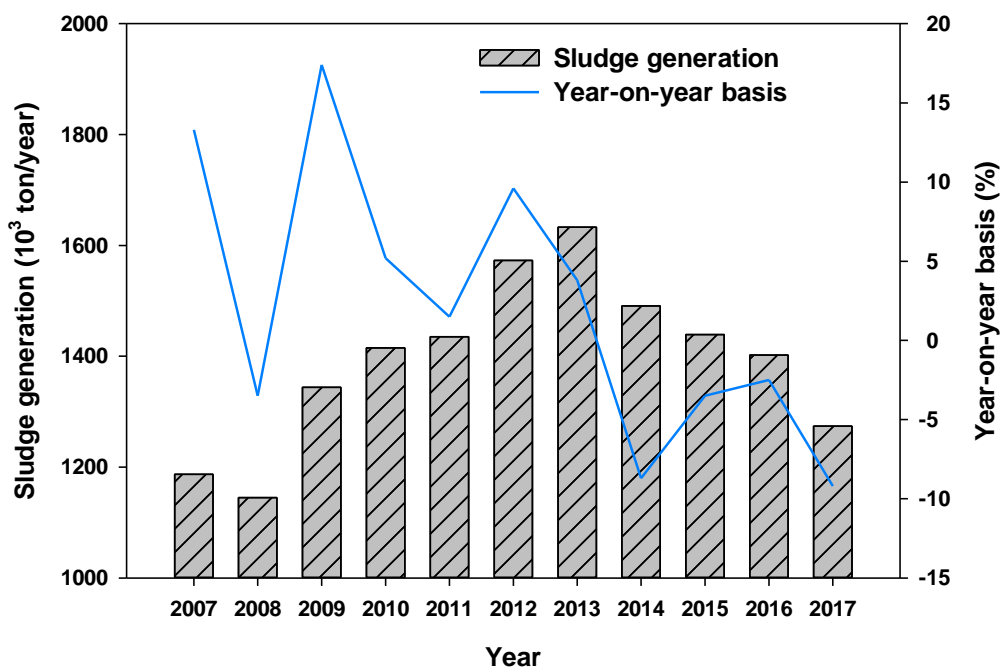


Fig. 1. Statistical data on sludge generation by year in Korea

This study focused on the synthesis of carbonates based on the minerals present in paper sludge. Moisture and short fibers within the sludge can be removed by drying, dehydration, and heat-treatment processes. By-products of the separation process can be recovered and used as raw materials for carbonate synthesis.

Heat-treatment technologies have been developed, including incineration, pyrolysis, steam reforming, and wet oxidation. Incineration exhibits a high energy consumption, but it can reduce the final volume of the sludge cake to 4% of its original volume, assuming a proportion of 20% total solids of the sludge cake (Task Force on Thermal Destruction 1990). Additionally, it is known that when an outlet temperature of an incinerator is 800 °C or more, the odor components are thermally decomposed. Furthermore, dioxin and furan-based compounds, which can occur due to incomplete combustion at 850 °C or more, can be destroyed (McKay 2002).

This study analyzed paper sludge discharged within Korea and explored its characteristics. Recycled fillers were manufactured by carbonation based on the results of the analyses and the applicability of the proposed technique was evaluated. This approach

demonstrates the advantage of controlling the particle-size distribution and the particle shape of the synthesized carbonate.

EXPERIMENTAL

Material

The paper sludge used in this study was obtained from four domestic paper mills in the Republic of Korea. The sludge comprised mixed sludge (PS) of coated white paper and coated color sludge (CS), OCC sludge (OS), and DIP sludge (DS). Quicklime (W), which was prepared by calcining and grinding domestic limestone, was used as a control (as shown in Table 1). Upon receipt, moisture content of each paper sludge sample was measured, and the residual ash content was analyzed according to ISO 1762. The dried samples were then held at 80 °C in an oven dryer for 24 h to prevent sintering. Next, the ash from the paper sludge was crushed once in a multi-purpose grinder (DSMP-370, Duksan, Gyeonggi-do, Korea). The pulverized paper sludge was calcined for 12 h and heated in a muffle furnace at a rate of 2 °C per min until the set temperatures of 525 °C, 750 °C, and 950 °C were reached.

Table 1. Summary of Samples and Treatment Stage Codes

Source	Product Type	Fine Paper, Coated Paper	Coating Color	Linerboard	Newsprint	Quicklime (W)
Sample Code	Sludge	PS	CS	OS	DS	-
	Sludge ash (Temp.*)	PSA _{Temp}	CSA _{Temp}	OSA _{Temp}	DSA _{Temp}	W
	Recycled filler	PF	CF	OF	DF	WF
*Note: Temp. represents temperature						

Methods

Physicochemical and thermal properties of paper-mill sludge and ash

The optical characteristics (L^* , a^* , b^* , and the ISO brightness) of the paper sludge samples were measured using a spectrophotometer (CM-2500d, Konica Minolta, Tokyo, Japan). The components were analyzed by XRF (ZSX Primus II, Rigaku, Tokyo, Japan). To analyze the thermal characteristics, mass loss upon heating was evaluated by increasing the temperature from room temperature to 1200 °C at 10 °C per min via TGA (SDT Q600, TA Instruments, Delaware, USA). The calorific value of the paper sludge was measured using an automatic isoperibol calorimeter (Parr 6400, Parr Instrument Company, Illinois, USA).

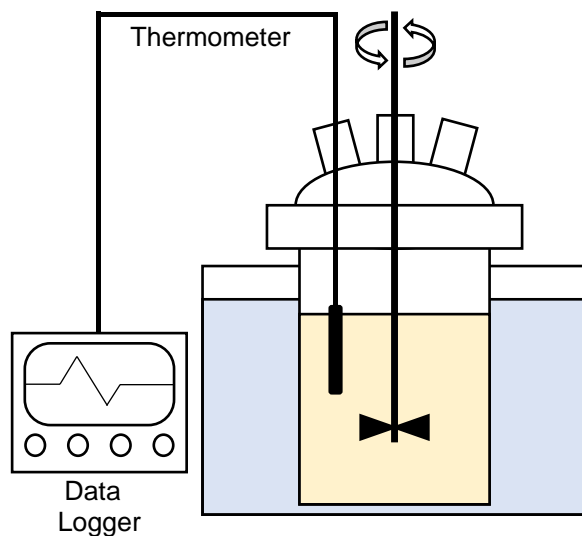


Fig. 2. Schematic diagram of the hydraulic activity of paper sludge ash used as a recycled filler

Ash was obtained by incinerating the paper sludge. The ash was evaluated according to the incineration temperature by measuring the chromaticity in the same way as the paper sludge. Evaluation of hydration activity based on the standard test method ASTM C110-02a (as depicted in Fig. 2) (You *et al.* 2017) was conducted to measure the heat of hydration of the paper sludge. In accordance with ASTM C110-02a, the hydration activity was evaluated by the difference between the hydration initiation temperature (T_0) and the temperature after 30 s (T_{30}). However, the activity of the paper sludge ash used in the study was lower than that of ordinary W. Therefore, the proposed standard was modified to evaluate the hydration activity based on T_{120} , which is 120 s later as calculated in Eq. 1.

$$\text{Hydration Activity} = T_{120} - T_0 \quad (1)$$

Manufacturing process and evaluation of the recycled filler

The paper sludge ash was subjected to a carbonation reaction, as presented in Fig. 3. The CO_2 gas was injected by MFC (mass flow controller). The end point of the recycled filler manufacturing reaction was defined as the point in which the pH was neutralized. The manufactured recycled filler was dispersed using an ultrasonicator before it was evaluated for particle-size distribution and average particle size by a particle-size analyzer (Mastersizer 3000, Malvern Ins. Ltd, Malvern, United Kingdom). The recycled filler was then dried at 105 °C for 24 h before being ground, using a mortar. The chromaticity was measured in the same way as for paper sludge and ash. The crystal lattice structure of the recycled filler was analyzed *via* XRD (X'Pert PRO MPD, PANalytical, Almelo, Netherlands). The morphology was observed *via* FE-SEM (JSM-7401F, JEOL Ltd, Tokyo, Japan) at a magnification of 30,000 times under an acceleration voltage of 5.0 kV.

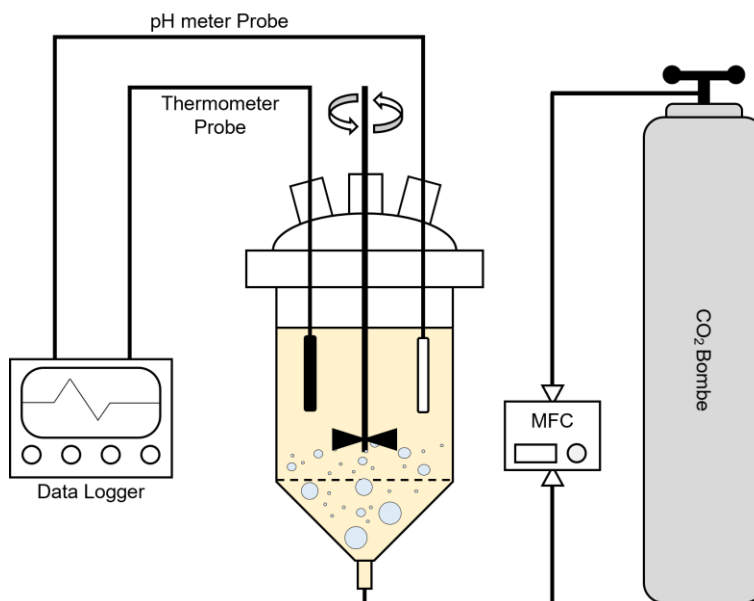


Fig. 3. Schematic diagram of the mixing batch system

RESULTS AND DISCUSSION

Properties of Paper Sludge and its Ash

The paper sludge samples were subjected to analyses of their moisture content, ash, and components (as presented in Table 2).

The Ca element in paper sludge is an important factor in determining the purity of CaCO_3 in manufactured recycled filler. Therefore, selecting and managing paper sludge with a high Ca content is necessary. Additionally, Mg may form $\text{CaMg}(\text{CO}_3)_2$ salt during the carbonation reaction with Ca ions, which can influence the degree of supersaturation of the hydrated slurry and affect crystal growth. Furthermore, it might be regarded as an impurity, as it does not participate in carbonation reactions, such as Si, Ti, Fe, *etc.* Colored components, especially those containing Fe ions, will result in a noticeable effect on the chromaticity of the paper sludge. Therefore, the Fe ion should be considered in terms of use or purpose.

Table 2. Moisture Contents, Ash, and Chemical Composition of Paper Sludge and W

Code	M.C. (%)	Ash (%)	Element Composition									
			C	O	Mg	Al	Si	S	Ca	Ti	Fe	Other
PS	55.0	52.5	14.8	52.5	0.73	2.26	2.19	0.357	25.6	0.077	0.682	0.682
CS	29.0	73.0	13.2	46.7	0.501	2.25	2.28	0.194	34.1	0.043	0.072	0.660
OS	50.9	71.8	ND	ND	0.65	5.43	3.19	1.16	42.5	0.157	40.8	6.113
DS	30.9	64.3	11.8	52.6	1.09	2.97	4.27	0.138	26.1	0.271	0.363	0.398
W	-	-	5.53	52.6	0.606	0.172	0.354	0.027	40.5	ND	0.10	0.111

*Note: ND means not detected

Consequently, two loss-of-mass sections occurred (as depicted in Fig. 4). The first was the section in which the decomposition of organic matter occurred. The second was the section where CO₂ stripping from CaCO₃ and the dehydration of kaolin minerals occurred.

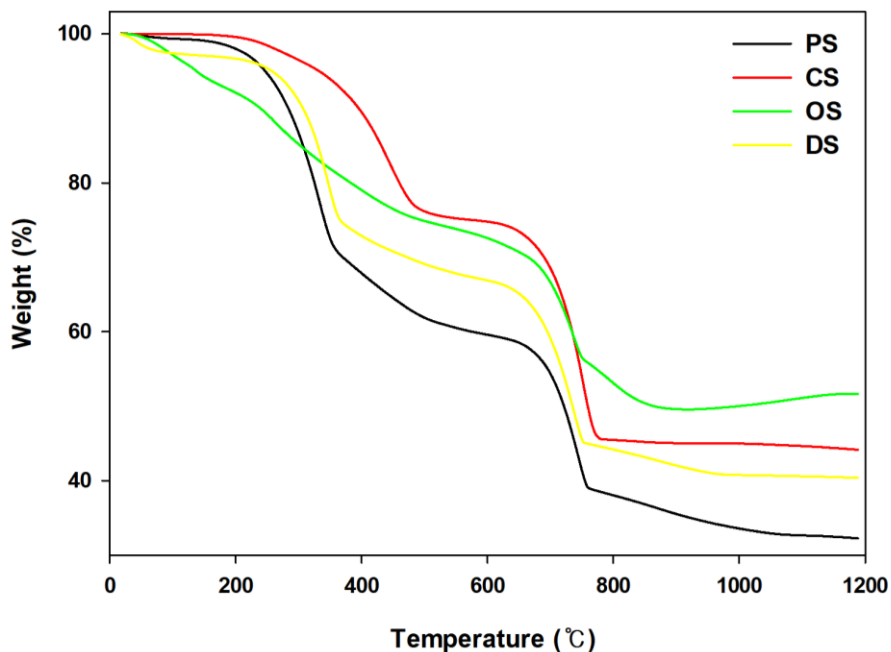


Fig. 4. Thermal analysis of sludge from different paper mills

In the case of the PS and DS, cellulose-based short fibers were decomposed in the first section, while in the coating color sludge (CS), styrene butadiene (SB) latex (not short fibers) was decomposed. Therefore, the loss curve was shifted to the right. Although the OS was modified through various treatments during sludge generation (despite the presence of short fibers), it was considered that the first loss section was not clearly identified. According to the results of Table 3, the high calorific value was evaluated in order of PS, CS, DS, and OS (Fig. 3). This seems to be proportional to the total amount of ash at 525 °C, except for the CS. The CS is not proportional, because the CS included the SB latex as a component in the coating color while the PS, OS, and DS all included short fibers.

Table 3. Gross Heating Value of Paper Sludge

Code	PS	CS	OS	DS
GHV (kcal per kg)	2198.17	1881.89	919.58	1751.78

The highest brightness values of the sludge were measured in the following order: CS, PS, DS, and OS. The PS, CS, and W were yellowish in color, the OS was brown-reddish, and the DS was greyish. In the case of the ash, the brightness increased in the order of ash calcined at 525, 950, and 750 °C, except for CSA (as presented in Table 4). The ash calcined at 525 °C exhibited a charcoal-greyish characteristic with low whiteness when compared with the other conditions. This characteristic occurred due to the presence of carbonized components from incomplete combustion. The ashes calcined at 750 °C

exhibited a slightly higher measured whiteness than the ashes calcined at 950 °C. Ash calcined at 950 °C caused CO₂ stripping, as the carbonate-type fillers were calcined and simultaneously became porous in structure (Potgieter *et al.* 2002). Consequently, the foreign matter and minerals that were mixed during the sludge-discharge process were exposed to the surface, which resulted in a slight reduction in whiteness. Conversely, the CS was directly recovered and used the sludge from the coating machine. Therefore, the coloring of the divalent iron salt due to the Fenton treatment sludge-discharge process was omitted and thus, the whiteness level appeared elevated. In addition, highly white PCC (not GCC) was used, while the binders, insolubilizers, dispersants, and defoamers used as additives were all decomposed *via* heat treatment, so it would not be affected to brightness level.

Table 4. Changes in $L^*a^*b^*$ and ISO Brightness Results from Paper Sludge, Ash, and W

Code	L^*	a^*	b^*	Brightness (% ISO)
PS	72.30	4.97	25.45	26.44
CS	87.96	1.99	16.92	54.72
OS	38.93	16.55	45.84	1.66
DS	60.44	-0.21	5.70	25.30
W	90.26	0.44	7.40	68.48
PSA ₅₂₅	64.51	0.93	3.54	31.07
PSA ₇₅₀	82.51	0.79	15.89	46.40
PSA ₉₅₀	79.34	0.43	20.78	37.99
CSA ₅₂₅	63.97	0.95	3.57	30.42
CSA ₇₅₀	92.21	-0.22	4.59	75.92
CSA ₉₅₀	94.53	-0.27	4.40	81.24
OSA ₅₂₅	42.73	22.13	56.95	1.21
OSA ₇₅₀	44.13	13.52	25.66	6.04
OSA ₉₅₀	37.16	13.83	32.17	2.67
DSA ₅₂₅	42.88	1.49	5.54	11.16
DSA ₇₅₀	89.15	0.74	6.23	67.58
DSA ₉₅₀	86.62	0.33	11.68	57.29

The hydration temperature was altered by the reaction time of paper sludge ash (Fig. 5). The hydration reaction of W was exothermic, and it is known that the rate of hydration can be predicted through the exothermic characteristic. Consequently, the initial hydration activity of PS was the highest, but it gradually decreased from the T_{60-80} periods.

Next, the high hydration activity was measured in the order of W, DS, and CS. The W showed lower initial activity than the PS, but activity continuously increased which might be attributed to the difference in raw materials. As OS is estimated to generate little heat, it is unsuitable for the manufacture of recycled fillers. Therefore, OS was excluded from the manufacturing of carbonate proceeds before the experiment.

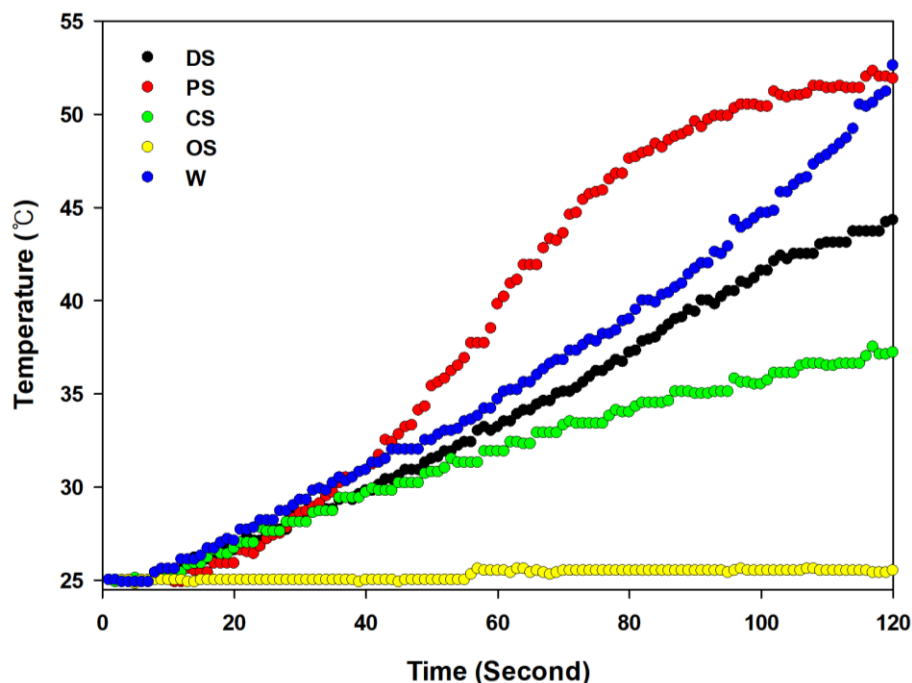


Fig. 5. The behavior of the heat liberation rate during the hydration of paper sludge ash from different paper mills

Crystallization and Characterization of Recycled Fillers

The chemical composition of recycled fillers is shown in Table 5. In the case of W at 18%, plugging occurred frequently both at the sensor and in the pipe. Thus, the manufacturing consistency was lowered to 15%. The chromaticity results of the manufactured recycled fillers exhibited no difference according to the amount of CO₂ injected (as given in Table 6). This is attributed to the sharing of the raw materials. The brightness of the recycled filler was measured to be higher than the ash calcined at 750 °C. This is because the Ca(OH)₂ in the hydrated slurry was precipitated by the carbonation process, while impurities and foreign matter cannot participate in the reaction. Therefore, such precipitation of impurities does not occur (Kemperl and Maček 2009).

Table 5. Chemical Composition of Recycled Fillers

Code	Element Composition									
	C	O	Mg	Al	Si	S	Ca	Ti	Fe	Other
PF	3.850	52.1	1.180	3.460	3.390	0.262	34.1	0.090	0.799	0.769
CF	4.460	53.1	0.523	2.180	2.250	0.104	36.5	0.043	0.075	0.765
DF	4.110	52.8	1.330	3.510	5.170	0.094	32.0	0.292	0.422	0.282
WF	5.430	53.2	0.634	0.196	0.373	0.033	39.9	ND	0.111	0.123

*Note: ND means not detected

The particle-size analysis results of the recycled fillers are presented in Fig. 6 and in Table 7. Under all the manufacturing conditions, tails are present in the particle size distribution area of less than 1 μm. The particles that were present in the form of single crystals without aggregation in the process of crystal growth were measured. Additionally,

the tails in the particle-size distribution area of over 10 μm were present under conditions other than just the WF. The particles that did not participate in the carbonation reaction, such as salts of the Al, Si, Ti, and Fe elements, were relatively high, as shown in Table 5. Additionally, the coagulated particles during the crystallization process were measured.

Table 6. Changes in $L^*a^*b^*$ and ISO Brightness Results by Conditions of Recycling Filler

Code	CO ₂ Injection (L per min)	Brightness (% ISO)	L^*	a^*	b^*
PF	0.3	70.76	93.16	1.77	10.61
	1.0	68.74	92.84	1.81	11.88
CF	0.3	95.95	99.12	-0.01	1.32
	1.0	95.79	99.05	-0.02	1.31
DF	0.3	77.10	94.01	1.12	6.72
	1.0	77.79	94.23	0.90	6.55
WF	0.3	91.02	97.66	0.30	2.32
	1.0	92.23	98.05	0.28	2.12

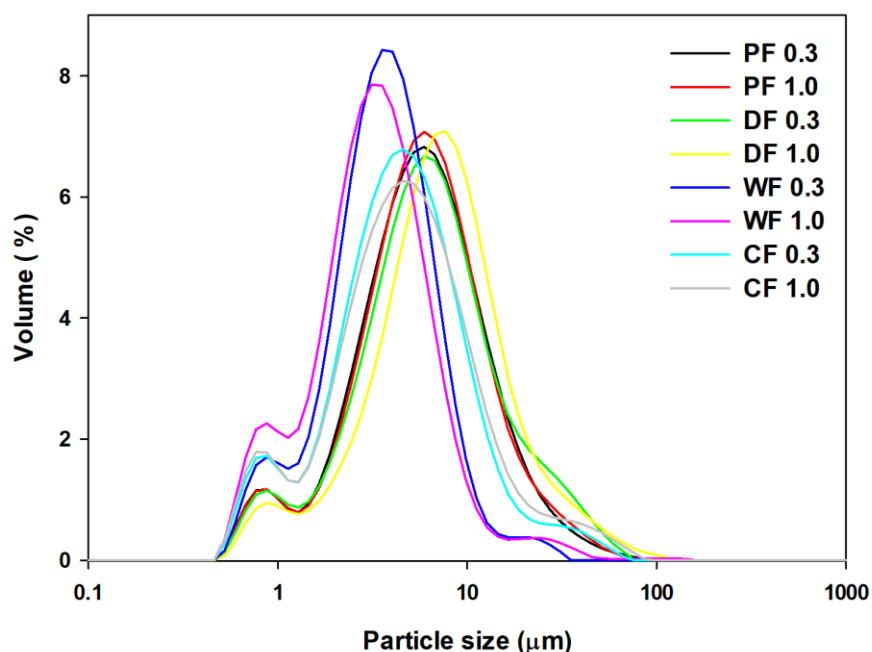


Fig. 6. Particle-size distribution of different recycled fillers

Table 7. Particle Size of Different Recycled Fillers

	PF 0.3 (μm)	PF 1 (μm)	DF 0.3 (μm)	DF 1 (μm)	WF 0.3 (μm)	WF 1 (μm)	CF 0.3 (μm)	CF 1 (μm)
D [4, 3]	8.26	8.3	9.57	10.7	4.62	4.65	6.74	7.66
d (0.1)	2.05	2.09	2.09	2.44	1.46	1.16	1.46	1.39
d (0.5)	6.08	6.14	6.5	7.58	3.81	3.39	4.69	4.91
d (0.9)	16.2	16.3	20.8	21.1	8.13	7.83	12.6	14.9

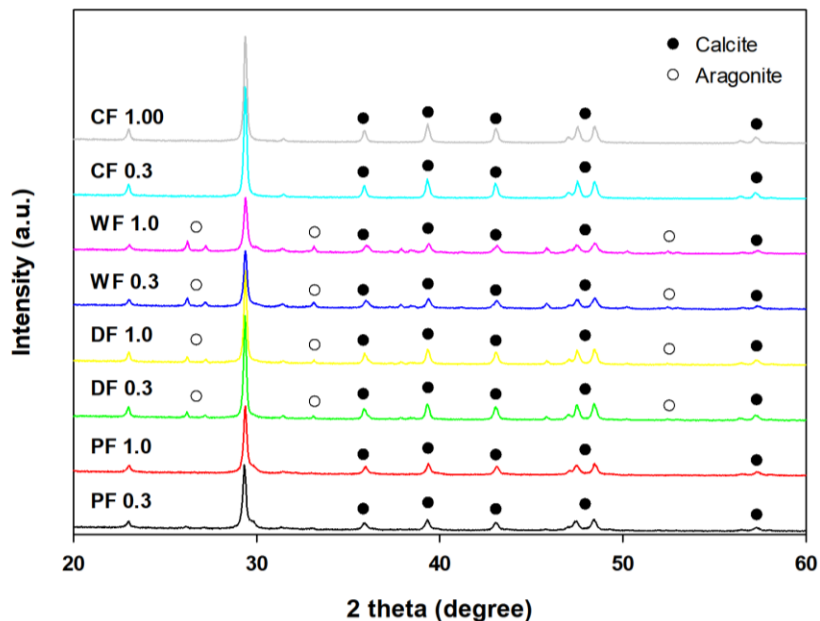


Fig. 7. XRD spectra of different recycled fillers

The average particle size was affected by the amount of CO₂ injected in the case of DF. As CO₂ was injected at a rate of 1.0 L per min, the carbonation reaction was promoted at more than 0.3 L per min. The temperature of the slurry in the mixing batch was increased. Consequently, the supersaturation of the hydrated solution was lowered, thereby promoting the synthesis of aragonite crystals with a large aspect ratio, as shown in Fig. 7. This contributes to an increase in the average particle size, as depicted in Fig. 6 and Table 7 (De Choudens-Sanchez and Gonzalez 2009).

Electron micrographs of the recycled filler are shown in Fig. 8, where rhombohedral crystals were observed in the case of the PF synthesized at 0.3 L per min. Most of the recycled fillers were observed in scalenohedral form, while the CF and DF were observed in the form of needle-like crystals. This might be because ions and impurities other than Ca influenced the crystals' growth mechanism (Meldrum 2003; Lin and Singer 2009; Wang *et al.* 2013).

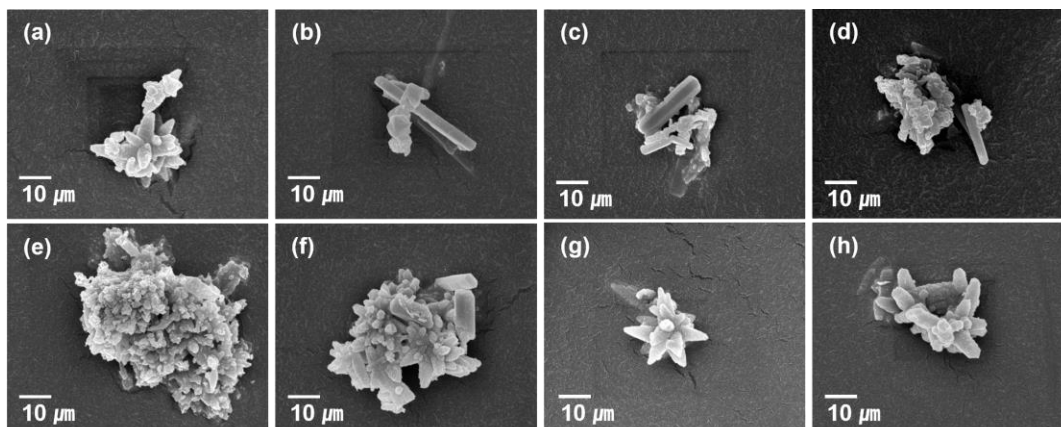


Fig. 8. FE-SEM images (a: CF 0.3 L per min, b: CF 1.0 L per min, c: DF 0.3 L per min, d: DF 1.0 L per min, e: PF 0.3 L per min, f: PF 1.0 L per min, g: WF 0.3 L per min, h: WF 1.0 L per min) under conditions of recycled filler

Generally, filler additives for paper dominated the scalenohedral form. Therefore, the CF, which exhibits a scalenohedral morphology and high brightness, can be used as a filler for PPC instead of commercial PCC. The DF is scalenohedral and needle-like, with an ISO whiteness of approximately 77%.

In conclusion, PF and DF could be used as an internal filler for papers that require reduced levels of whiteness, such as eco-friendly recycled copier papers in which de-inked pulp is used. In addition, PF and DF exhibit slightly lower whiteness levels than CF. They are expected to replace some of the market demand in various industries that do not require highly white PCC levels considering the crystal morphology.

CONCLUSIONS

1. Paper sludge ash generated as an incineration by-product could be used as a source of calcium to use in the preparation of CaCO_3 filler, to be used in papermaking. For the manufacture of recycled fillers, the only added ingredients were water and CO_2 .
2. An inorganic element analysis revealed that the percentage of Ca decreased in the order of old corrugated container mill sludge (OS), coated paper sludge (CS), deinked paper sludge (DS), and mixed sludge (PS). However, the results of hydration activity showed that CS was somewhat low in activity, while OS was very low. Therefore, OS was deemed to be unsuitable to produce carbonates.
3. Recycled fillers can be compared with commercial precipitated calcium carbonate (PCC) with similar manufacturing mechanisms. In terms of brightness and morphology, CF exhibited a similar brightness and form to conventional paper-based fillers, with the potential to replace commercial PCC. Conversely, PS and DS were somewhat low in brightness but were considered morphologically acceptable as papermaking fillers.

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