Research paper

Disposal of Solid Wastes from the Paper Industry Using a CHTC-based Technology: Optimization of the Treatment Conditions

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【Summary】

In a previous study, we explored the feasibility of applying the catalytic hydrothermal conversion (CHTC) technique in treating organic wastes from pulp and paper mills. In the present study, we focus on optimization of the conversion process based on sludge and process rejects of a wastepaper-based whiteboard mill. Under a 70% moisture content, heating to a reaction temperature of 750℃ with 10 g of oven-dry (o.d.) sludge, an equal part heating medium (sericite) and ~5% sodium carbonate catalyst produced 61% hydrogen in the flue gas (syngas) for a total hydrogen yield of 3770 mL. Under the best conditions, the same quantity of coarse-particle process rejects, on the other hand, produced 42% hydrogen in the syngas for a total hydrogen yield of 3173 mL. The ANOVA results of a 2^4 factorial design using solid waste particle size, catalyst dosage, heating medium amount, and reaction temperature as variables indicated that the interactions of heating medium amount and temperature in both the sludge and process reject groups were statistically the most significant. This reflects the importance of heat conduction efficiency in the current reactor design and the need to improve future scale-ups. The main effects of the catalyst and heating medium were both positive, suggesting that the choices for the current experiments were effective. The particle size and reaction temperature factors exhibited opposite effects in the sludge and process reject groups, probably reflecting variations in organic species and contents in the 2 groups.

Key words: CHTC (catalytic hydrothermal conversion), solid wastes, disposal, energy recovery, volume reduction.

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研究報告

紙業固體廢棄物催化劑濕熱轉化之最適化研究

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摘 要

國內漿紙工業的困擾之一為隨廢紙原料等夾雜而來的固體廢棄物處置問題。本研究擬採新穎之催化劑濕熱轉化法處理以解決此問題。由某白紙板廠收集之紙業固體廢棄物包括污泥與排渣兩大類，分別予以定性。處置試驗取10 g絕乾之試材，添加近等量之熱傳導助劑(絹雲母)及約5%量之碳酸鈉催化劑於含水率70%之條件下以第二型反應器加熱至750℃，以及約50 min之反應時間處理。污泥部份之試驗結果顯示於750℃添加50%熱傳導劑與5%碳酸鈉條件下可獲得61%之氫氣生產率及3770 mL之氫氣。排渣部份試驗則顯示粗渣於750℃、30%熱傳導劑與5%催化劑條件下最高可獲42%之產氫率及3173 mL之氫氣。以材料粒徑、催化劑量、熱傳導劑量與反應溫度為參數之2^4階乘試驗，污泥與排渣處理的ANOVA分析中顯示熱傳導劑與溫度的交互作用統計上最為顯著，突顯目前反應器熱傳導效率的良窳對CHTC反應影響最顯著，而如何改善反應器設計以提高熱傳導效率為未來加大型之主要課題。催化劑與熱傳導劑的主效應均呈正效應，則說明目前選材正確；至於溫度與材料粒徑在污泥與排渣組皆呈相反之效應，則可能與試材有機質的種類與含量變異有關。

關鍵詞：催化劑濕熱轉化、固體廢棄物、處置法、回收能量、減容。

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INTRODUCTION

Traditionally, Taiwan's paper industry has relied heavily on recovered or secondary fiber resources due to a lack of indigenous wood and fiber supplies. The local wastepaper recovery rate and utilization rate are quite high among nations of the world. Despite the good connotation of utilizing resources more fully and being environmentally friendly, extensive practices based on wastepaper also have their downside, in addition to inferior fiber quality and operational problems, non-fiber substances in the recovered wastepaper cause big headaches for paper mills, as these entrained foreign matter can make up 10~20% of the wastepaper, and their diverse natures require various unit processes such as screening and centri-cleaners to remove them from fiber stocks. The accumulated rejects and the bio-sludge arising from wastewater treatments thus represent a heavy burden for the industry in terms of disposal. There is not much one can do about the heavy contraries, such as sand, pebbles, glass shards, metals, etc. The lighter fractions often include organic substances such as styrofoam, plastic string, latex gums, etc. that can be disposed of through incineration or landfilling (Shen and Chang 2001). Better means of disposal for such substances are sorely needed. The American Forestry & Paper Association (AF&PA) wrote a future technological platform with a bold outlook to resolve problems facing the wood-based industry in 1994. It is called *Agenda 2020* and one important plank of the agenda is to develop the integrated forest bio-refinery (IFBR) mainly by applying gasification technologies to pulp mill black liquors and forest residues, substituting the traditional recovery boiler system and modifying the recaustization process to generate syngas in the process and enhance the energy recovery of the industry as well as supply excess energy to the general economy (Raymond and Closset 2004). The concept is akin to what we are trying to achieve, that is to explore the potential of a viable gasification process to convert organic residues of the paper industry into useful fuel and minimize the waste volume in the process. Based on a blueprint supplied to us by the Applied Research Associations, Panama City, FL, USA (Renard and Li 2003, personal communication), and with their authorization, we tested a bench-top catalytic hydrothermal conversion (CHTC) reactor to treat sludge and process rejects of a local whiteboard mill.

Unlike other well-known processes such as wet-oxidation (WO), supercritical wet-oxidation (SCWO), and hydrothermal processes (HTP), that often require exceedingly high pressures or reaction temperatures, CHTC entails using water steam to react with organic molecules and generate hydrogen and carbon monoxide gases in a mid-temperature range (usually between 650 and 850℃) and at atmospheric pressure. Commonly used catalysts are either carbonate salts of alkaline metals (sodium or potassium) and activated metal substrates (nickel, in particular).

In the study, we employed a 2^n factorial design using solid wastes of different grain sizes, catalyst dosages, heating medium amounts, and reaction temperatures as variables. The exit gases of the reaction products were collected and their hydrogen gas contents analyzed. The condensates and solid residues were separated and analyzed as well to determine the optimal conditions for carrying out the treatment. A new bench-top reactor auger with a 1.5-in (3.8 cm) bore dimension was used, with the rest of the setup largely identical to the first-generation reactor used in our previous study (Wang et al. 2005).

MATERIALS AND METHODS

Materials

The sludge from the mill wastewater treatment facilities and process rejects of the mill screen units of a whiteboard producer, Kuan-Yuan Paper, in Tachia Township, Taichung, central Taiwan were collected for the study. Industrial-grade sodium carbonate as the catalyst and coarse sericite (with an average grain size of 14 μm, and a bulk density of 2.6 g/cm^3) supplied by Sun Mica, Taitung, Taiwan as the heat conducting medium were used.

Methods

Analyses and preparation of sludge and process rejects

Samples were air-dried and then Wiley-milled to pass through screens of specific mesh sizes. Three grain sizes resulted and were designated as fine (f), medium (m), and coarse (c). The initial moisture content and volatile organic content (by ashing at 575℃) of the particles were then analyzed separately. The mill process rejects being rich in adhesives and plastics were difficult to mill. We used dry ice to cool and harden the substances during milling.

Catalytic hydrothermal conversion treatments

The experiment was carried out using a 2^4 factorial design with particle size, reaction temperature, catalyst dose, and heating medium amount as the variables; this entailed 16 combinations of experimental conditions and 3 replications of the center point (Table 1). The sludge and process reject samples of different grain sizes were adjusted to a target moisture content of 70±2%. Portions of 10 g o.d. specimens were added with sodium carbonate catalyst at 3 and 5% (4% at the mid-point), and sericite heating medium at 30 and 50% (40% at the mid-point). The thoroughly mixed specimens were transported by an auger rotating at 1 rpm into an upwardly inclined reactor preheated to 750 and 800℃ (775℃ at the mid-point). The reactor was similar to the first-generation reactor we used in the previous study, except having a larger tubing with inside diameter of 38 mm (1.5 in) SS310 stainless steel and a length of 75 cm, and the auger had a screw pitch of 1 in (2.54 cm). Outside of the reactor tubing, there were 2 heating jackets encircling it that can provide a reaction temperature of up to 1,000℃; the heaters were controlled by a feedback loop of thermal couples. The CHTC reaction then passively took place without a separate injection of steam. The exit gases from the end of a condenser and gas/liquid separator were collected and their hydrogen gas content determined. The ashes and liquid condensates of the reactions were also collected and their COD values analyzed.

Table 1 shows the experimental designations of the factorial design and the run numbers corresponding with the design. In practice, the run numbers were executed randomly to avoid systematic errors.

Table 1. Parameters in the 2^4 factorial experimental design with normalized coefficients and the run number designations

Code		-1	0	+1
P (particle size)		coarse	medium	fine
C (catalyst)		3%	4%	5%
M (medium)		30%	40%	50%
T (temperataure)		750℃	775℃	800℃
Run no.	P	T	C	M
1	-1	-1	-1	-1
2	1	-1	-1	-1
3	-1	1	-1	-1
4	1	1	-1	-1
5	-1	-1	1	-1
6	1	-1	1	-1
7	-1	1	1	-1
8	1	1	1	-1
9	-1	-1	-1	1
10	1	-1	-1	1
11	-1	1	-1	1
12	1	1	-1	1
13	-1	-1	1	1
14	1	-1	1	1
15	-1	1	1	1
16	1	1	1	1
17	0	0	0	0
18	0	0	0	0
19	0	0	0	0

RESULTS AND DISCUSSION

CHTC reactions

When organic-containing solid wastes undergo heating in the absence of oxygen they produce gases, liquids (tars), and solid residues (coke and ashes). When steam is injected at high temperatures, however, steam reformation and water-gas shift reactions occur, particularly with the help of catalysts leading to the production of syngas composed mostly of hydrogen and carbon monoxide. The tar and coke production is largely eliminated (Buekens and Schoeters 1985). The reaction products are functions of the reaction temperature, the catalyst used, the steam-to-carbon ratio, etc. Carbonate of alkali metals and supported nickel are effective catalysts capable of lowering reaction temperatures and minimizing the solid- and liquid-phase products (Fung and Graham 1980).

Parameters of sludge and process rejects from the Kuan-Yuan mill site

Table 2 shows the air-dried moisture contents (MCs) and the volatile (organic) fractions of the sludge and process rejects of different grain sizes from the Kuan-Yuan whiteboard mill. The initial MCs of the materials as collected from the mill are also shown. The sludge sample was relatively homogenous with respect to the organic content, whereas the process rejects of different grain sizes often had disparate organic contents, probably due to the preferential distribution of different organic components. Overall, the finer the grain sizes, the lower the organic content was, indicating that inorganic components were probably mostly fine particles that tended to accumulate with the finer fractions during milling and screening. The influence of particle size distribution on the CHTC reaction was tested using an ANOVA based on the 2^4 factorial design.

Usual methods of treating solid wastes from the pulp and paper industry entail the steps of concentrating, stabilizing, dehydrating, and then disposing through sanitary landfilling, incinerating, or spreading these materials on fields as ground-ameliorating agents and composting (Hynninen 1998). Most of these methods are difficult to apply in Taiwan, where the land area is limited and population density is very high. Furthermore, as shown in Table 2, the sludge and process rejects accrued at various mills had a wide range of variations that could make their disposal more difficult. Characterization of Taiwan's paper industry solid wastes was carried out by Shen and Chang (2001) which concurred with the above statements. Thus, novel and applicable solid waste disposal technology shall be a boon to the industry.

Solid-phase products

As shown in Fig. 1, typical reactions of the sludge and process rejects at 575℃ produced less than 10% volatile residues. Organic substances in the solid wastes had conversion rates of 85~95%. The results were somewhat inferior to what was achieved during the first series of experiments (Wang et al. 2005), the reason was mainly the poorer interior heat conduction due to the larger reactor tube size. As will be noted from observations of the overall results, the reaction was deemed to be constrained by heat transfer and required longer reaction times or better means of mixing and/or heating. Figure 1 shows that unconverted volatile organics made up 5~15% of the ashes after the CHTC reaction. Process rejects tended to be less homogeneous and poorer conversion materials than was the sludge. Figure 2 shows that ashes after the CHTC still made up about 40~60% of the original feed mass on an oven-dry basis. As a reflection of their respective inorganic contents, process rejects tended to

Table 2. Air-dried moisture contents, and volatile fractions of the sludge and process rejects of a whiteboard mill

Item	Initial MC (%)	Air dried MC (%)			Organics (%)		
Sludge	unsorted	c	m	f	c	m	f
	71.59	8.01	10.07	7.09	63.40	61.96	60.85
Process rejects	unsorted	c	m	f	c	m	f
	69.51	6.76	8.37	5.17	76.15	71.17	63.52

c, coarse; m, medium; f, fine.

be less treatable than the sludge from the mill. The results also suggested that the auger-type reactor using a heat transfer medium might have practical limitations in scaling up to a practical operational unit. One possible solution is to use active high-temperature steam injection into the reaction zone to assist in mixing and heat transfer. Perhaps a totally different design such as using fluidized-bed mixing should be considered.

Liquid-phase products

Most of the unconverted organic vapors should recondense upon cooling and be entrapped in the condensates of the gas-liquid separator. The pollution loadings of the condensates were analyzed. Figure 3 shows that substantially higher COD contents were observed for the condensates than in the previous bench-top trial (Wang et al. 2005), suggesting that the reactor length or the reaction time was probably insufficient for the complete conversion of the organic pyrolyzates, thus leading to the presence of tars in the condensates. The system controls allowed no slower setting for the rotating auger, however, making the problem an intractable systematic one. In general, sludge tended to produce greater amounts of tars than did the process rejects. The sludge might have contained more-oxidized organics which were less efficient in gasification, while the process rejects might have been richer in hydrocarbons and were thus gasified better.

The ANOVA results of the condensate COD values indicated that for the Kuan-Yuan sludge, the mean of the center point repeats (4105) was reasonably close to the grand mean of the experimental runs (3982), which indicates that a linear relationship probably exists for the experimental variables. Among the main effects, only the catalyst dosage (-1381) showed a strong negative correlation, indicating that increasing catalyst should help reduce the generation of tar in the condensate. For the interactions, P×C showed a strong effect (1103).

The ANOVA analysis of the condensate COD values for the mill process rejects showed that the mean of the center point

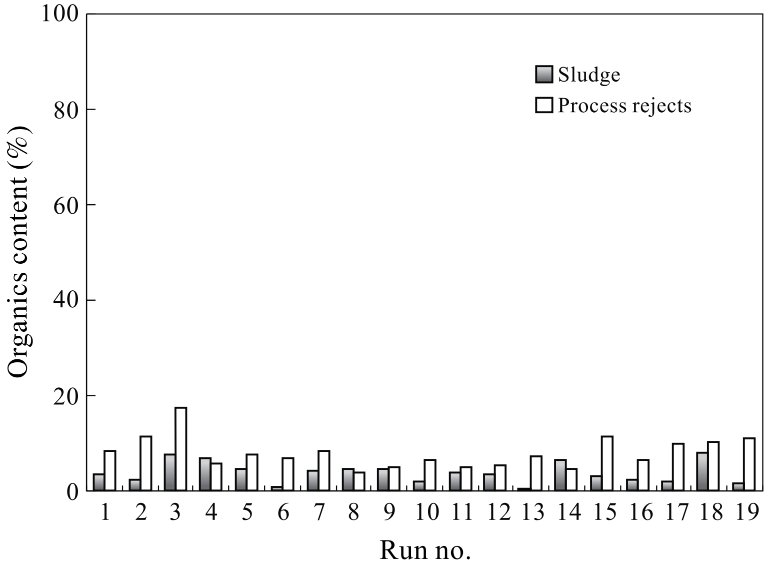

Fig. 1. Volatile contents of residues from sludge and process rejects in the CHTC runs.

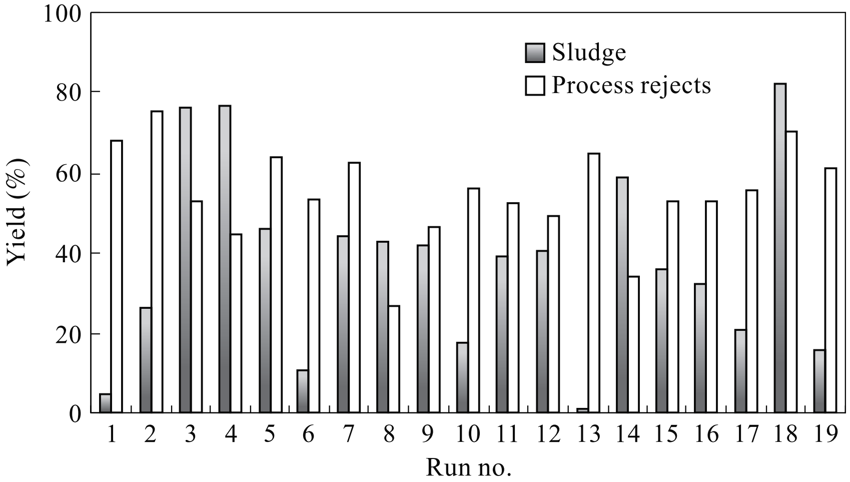

Fig. 2. Solid residue yields after CHTC treatment of the sludge and process rejects. The horizontal axis denotes runs which correspond to the run numbers in Table 1.

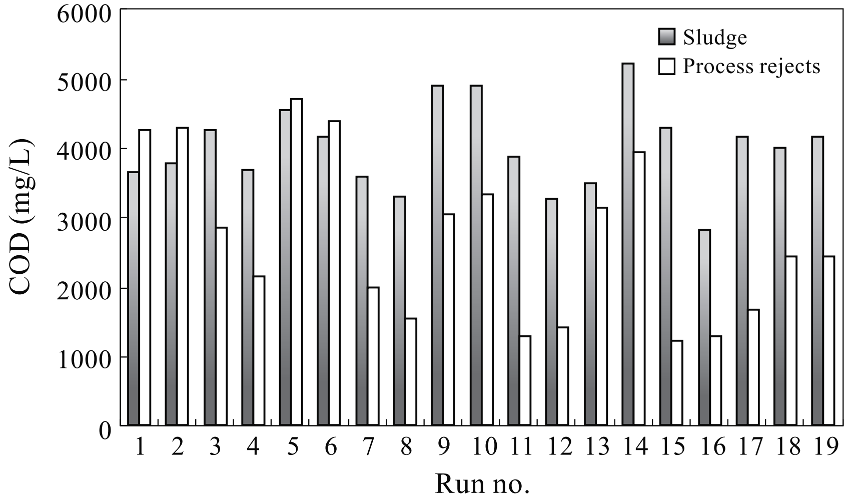

Fig. 3. COD values of the condensates collected after CHTC treatment of sludge and process rejects. The horizontal axis denotes runs which correspond to the run numbers in Table 1.

repeats (2172) distinctly differed from that of the experimental runs (2797), indicating that there was no linear correlation among the variables. Further interpretation of the results was not warranted. It is interesting to note, however, that the catalyst dosage exhibited a very strong main effect (-4316), followed by temperature (-1891), while most of the interactions were relatively weak. Thus, by increasing the catalyst and reaction temperature, less tar should be generated.

Gas-phase products

The main purpose of the CHTC treatment is to derive combustible syngas fuel containing hydrogen and carbon monoxide, thus recovering the energy content of the wastes and reducing the volume of the wastes for easier final disposal. Hence, gas-phase products are of prime importance. When an experimental run was begun and the system reached thermal equilibrium, the off-gas was collected using inverted PET bottles in a water trough. The hydrogen concentration of the collected gases was then measured using a hydrogen gas analyzer. As hydrogen gas is nature's smallest and lightest gas molecule, easily escaping from the smallest fissures, the gas-tightness of the reactor and collecting system could not be guaranteed. A certain fraction of the gas might thus have not been recorded. We tried to minimize errors in this regard by keeping the run times constant. Figure 4 shows the hydrogen content of the exit syngas from the reactor. For paper mill sludge, hydrogen gas made up around 40~60% of the syngas, whereas for the paper mill process rejects, hydrogen gas was only 30~40% of the syngas fractions. Volumes of the hydrogen gas generated as calculated from the volume fraction of the hydrogen concentration are shown in Fig. 5. Hydrogen gas generated in a single run ranged from ~2300 to 3800 mL, with a maximum of 3772 mL for the paper mill sludge and ~2000 to 3200 ml with a maximum of 3173 mL for the paper mill process rejects. Normally, the reaction rate depends on the original organic content, and the type of organics (Table 3).

Several anecdotal observations based on the experiments were made. 1) Hydrogen gas emissions from the sludge had good predictability. For instance, at a medium-to-catalyst (M/C) ratio of 10, the small-particle-size sample produced more hydrogen. At the other ratios, gas generation by small particles was inferior. Under certain circumstances, smaller particles of the process rejects performed better than did larger particles, whereas in general, larger process rejects performed better. 2) Hydrogen gas concentrations generated by the sludge and process rejects notably differed, with the former producing higher concentrations of hydrogen in the reaction. The total syngas volume productions from process rejects were higher and faster. 3) With higher sericite medium in the mix, the tar in condensates tended to decrease. This observation was congruent with the results for the liquid-phase products above. As the reaction was probably heat transfer-limited, use of a higher heat-conducting medium thus helped to lower the generation of tar in the condensate.

ANOVA of the experimental parameters and interactions

Table 4 and Fig. 6 present the ANOVA results of the 2^4 factorial experiments for the paper mill sludge. The signs of the coefficients suggest the direction of the influence, for instance a negative coefficient indicates that when the parameter goes from -1 to +1, it contributes to a reduction in hydrogen gas formation, and vice versa. The absolute value

Table 3. Summary of hydrogen generation by CHTC reactions of the paper industry sludge and process rejects

	Sludge	Process rejects
H_2 range (%)	40~60	30~40
Max. H_2 generation (%)	60.9	42.1
H_2 range (mL)	2300~3800	2000~3200
Max. H_2 generation (mL)	3772.3	3173.0

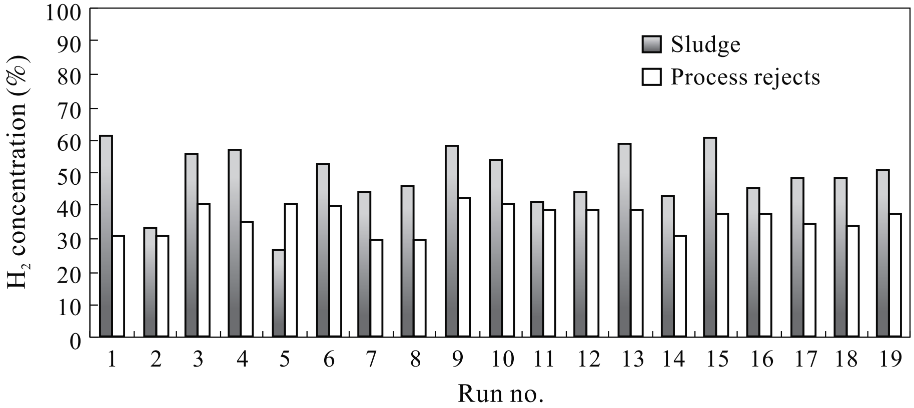

Fig. 4. Hydrogen gas yields (H_2, %) based on off-gas production collected after CHTC treatment of sludge and process rejects. The horizontal axis denotes runs which correspond to the run numbers in Table 1.

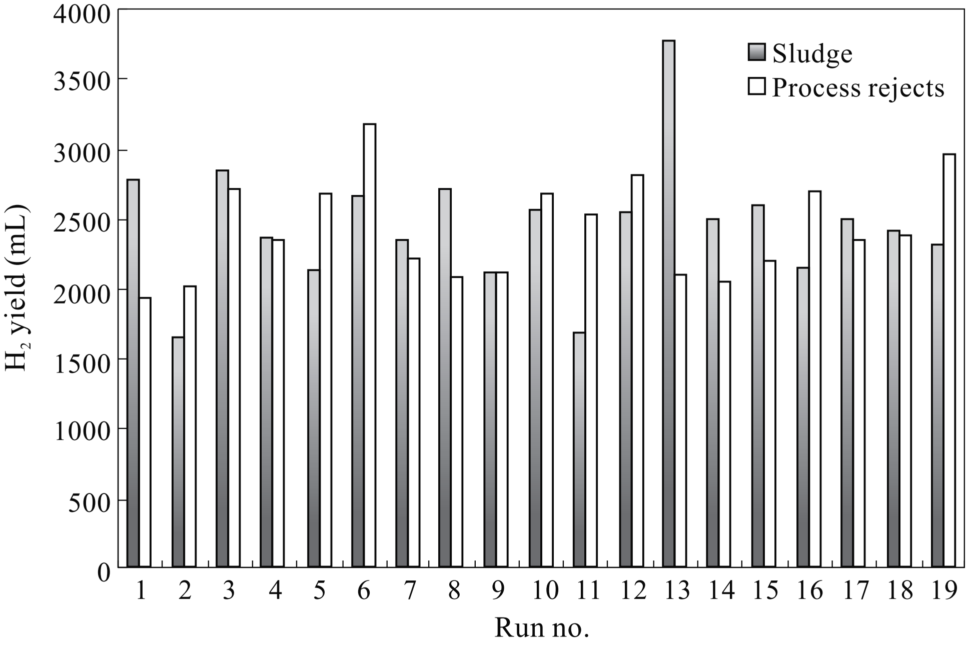

Fig. 5. Hydrogen gas volume (H_2, mL) collected after CHTC treatment of sludge and process rejects. The horizontal axis denotes runs which correspond to the run numbers in Table 1.

of the coefficient, meanwhile gives the magnitude of the influence exerted by the parameter in the range of observation.

The ANOVA results for sludge indicated that among the main effects, the catalyst (294), heating medium (376), and reaction temperature (118) were positive. Thus, the results suggest that in future operations, increasing the catalyst, medium, and temperature should have positive contributions to hydrogen gas generation. The particle size effect (-273), however was negative, suggesting that coarser particles performed better and that compositions of different size fractions might have varied results. Compared with the interactions, however, these were all statistically insignificant. The statistically significant interactions were sorted and are given here: M×T (-841), P×M (414), P×T (411). The remaining of interactions, when compared with the main effects, were insignificant. The means of 16 runs averaged 2470.4 mL hydrogen which was close to the average 3 median replications of 2407 mL hydrogen, thus suggesting that within the experimental ranges, the variables had linear relationships. Among the ANOVA results, the interaction of heating medium and temperature had a negative effect of the highest statistical significance; this illustrates that heat conduction efficiency influenced with the performance of CHTC reactions in the current reactor design. The statistical significance of the P×M and P×T interactions suggested that organic content differences among the particle size fractions should have notable influences on the CHTC reactions as well.

The ANOVA analysis of process rejects for hydrogen gas generation in the CHTC treatment runs are shown in Table 5 and Fig. 7. The results showed that the mean effects of particle size (474), catalyst (835), and heating medium (641) were positive for hydrogen generation. This suggests that in future operations, these parameters should be further increased, whereas, the temperature effect (-622) was negative, which might have had something to do with the organic content of different-sized fractions. Compared with the interactions, all the main effects were significant. Among the interactions, only M×T (-1193) was statistically significant, while all the remaining interactions were not significant compared to the main effects. The mean of the 16 experimental runs averaged 2563.3 mL hydrogen which was very close to the mean of 3 median replications of 2549.8 mL, suggesting that all of the variables had linear relationships within the experimental ranges. The results of ANOVA analysis for the process rejects were similar to those of

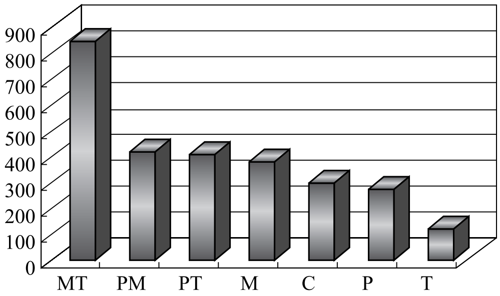

Fig. 6. Ranking of significant parameters and interactions for hydrogen generation of sludge in CHTC treatments.

Table 4. ANOVA results of the hydrogen generation from the sludge of a whiteboard mill

Mean H_2 (mL)	2470.4	Central mean H_2 (mL)	2407.0
P (size)	-273	C×T	-48
C (%)	294	M×T	-841
M (%)	376	P×C×M	-74
T (℃)	118	P×C×T	-164
P×C	-40	P×M×T	-19
P×M	415	C×M×T	156
P×T	411	P×C×M×T	72
C×M	-77		

Table 5. ANOVA results of the hydrogen generation from the process rejects of a whiteboard mill

Mean H_2 (mL)	2549.8	Central mean H_2 (mL)	2563.3
P (size)	474	C×T	-197
C (%)	835	M×T	-1193
M (%)	641	P×C×M	289
T (℃)	-622	P×C×T	209
P×C	-83	P×M×T	-384
P×M	186	C×M×T	82
P×T	188	P×C×M×T	130
C×M	3		

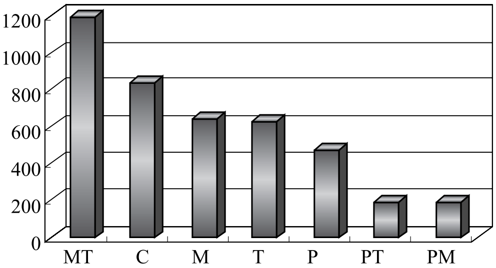

Fig. 7. Ranking of parameters and interaction for hydrogen generation of process rejects in CHTC treatments.

the sludge. The interaction of M×T also had the highest statistical significance and was negative as well. This again illustrates that for the present reactor design, the heat conduction efficiency controlled the CHTC reaction performance. Determining ways to enhance the heat conduction efficiency in a new reactor design would be the most-important task for the scale-up of the lab unit.

CONCLUSIONS

Following an initial study indicating that CHTC treatment of paper mill organic solid wastes can produce hydrogen-rich syngas, we proceeded to study various parameters which influence syngas production using a factorial experimental design. The results suggest that due to poor heat conductivity, the medium for heat transfer played a critical role in the passive mode of heating water-containing solid wastes for gas conversion. The maximum amounts of hydrogen gas generated by 10 g o.d. paper mill sludge was 3772 mL producing a 61% hydrogen gas concentration in the syngas. Those values for paper mill process rejects were 3173 mL and a 42% hydrogen gas concentration. A factorial design using solid waste particle size, catalyst dosage, heating medium amount, and reaction temperature as variables showed that generally, the catalyst and heating medium contributed positively to hydrogen generation; whereas the interaction of heating medium and temperature had the greatest statistical significance, suggesting that for the current reactor design, heat conduction efficiency may be the most-important controlling factor for CHTC reactions. The main effects of waste particle size and reaction temperature were opposite between the sludge and process reject groups, indicating that the organic composition and content variations of different-sized fractions of the 2 groups probably influenced the hydrogen gas generation outcomes.

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