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Simultaneous Microwave/Ultrasound-Assisted Hydrolysis of Starch-Based Industrial Waste into Reducing Sugars

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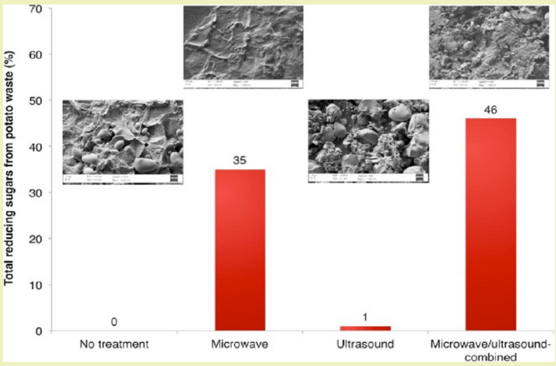
ABSTRACT: Catalytic conversion of starch-based industrial waste into sugars was investigated using a simultaneous microwave/ultrasound-assisted procedure. The results were compared with single microwave and ultrasound irradiations to determine the eventual synergetic effects generated by the combined technologies. In heterogeneous systems, microwave irradiation enforces heat transfer, while ultrasound irradiation imposes the intensive mass transfer at the interfaces. Two hours of combined irradiation of potato starch (employed as a reference) at 60 °C in sulfuric acid revealed the existence of a glucose selectivity. In a result that was hitherto never achieved, 46% of this industrial waste was converted into reducing sugars in identical conditions.



KEYWORDS: *Biowaste, Microwave/ultrasound-assisted hydrolysis, Carbohydrates, Starch, Reducing sugars*

INTRODUCTION

The intensive consumption of fossil fuels as a source of energy has brought about some global problems such as the greenhouse effect. However, the recent focus on renewable energy has begun to contribute to a reduction in this dependence upon fossil fuels. Current studies have concentrated on preempting the issues generated by this overconsumption, and although demand is intense, the production of biofuels might partially solve these issues. However, the origin of the transformable biosource should not compete with food, and waste should be subsequently privileged. On the plus side, biofuels have much undiscovered potential that is waiting to be investigated. For example, Jepuan Peruna Oy, a Finnish company producing ready-cooked vacuum potatoes for professional catering, generates about 20 tonnes of potato peel daily. Currently, 90% of this sludge waste is transported to cattle and pig farms, while the remaining 10% is transported to a waste dump for fertilizer and soil improver production. Total hydrolysis of the potato peel revealed that 88% could be subsequently considered as sugar potential. The main carbohydrate in potato peels is starch, composed of amylose and amylopectin, which are glucose building blocks. Glucose is recognized as a precursor for platform molecules in chemicals and fuel alcohol production.[1,2] In a previous paper,[3] we studied the hydrolysis of this nonwater-soluble biopolymer into reducing sugars under single nonconventional activation methods. The use of ultrasound and microwave technologies appealed to be appropriate, rapid, safe, and sustainable routes to depolymerizing starch into sugars. The depolymerization parameters were optimized under either microwaves or ultrasound (with distinctive phenomenon occurring at low and high frequency) irradiation and compared to basic mechanical stirring.[3] Even though the results were encouraging, each nonconventional activation method revealed limitations caused by the very complex matrix found in this type of waste. To overcome these limitations, we decided to combine the two energy sources. A combined device composed of a synthesis microwave and a low frequency ultrasonic probe might not only enhance the depolymerization rate but also emphasize any synergetic effect. Microwave irradiation is a rapid heating method for polar media that enhances selectivity, improves reaction rates, shortens reaction time, and reduces the number of side products.[4] The application of an alternative electric field provokes intermolecular friction between polar molecules, thereby releasing kinetic energy in the inner media by rapid heating.[5] In parallel, the passage of a low frequency ultrasonic wave through a liquid generates cavitation bubbles. This phenomenon, known as acoustic cavitation, is the formation, growth, and collapse of highly energetic microbubbles. During the collapse of these microbubbles, extreme conditions of high temperatures and pressures release shockwaves into the reaction medium, generating fragmentation of molecules. The implosion leads to specific effects capable of breaking down chemical bonds. Cavitation can result in chemical and mechanical effects according to the system and the frequency employed. The low frequency of ultrasound is beneficial to produce dominant physical effects as compared to chemical effects at higher frequencies. In heterogeneous systems,

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cavitation is mainly a mechanical effect. In a solid/liquid system, collapse of cavitation bubbles near the surface generate a liquid jet increasing mass transfer by disrupting the interfacial boundary layers and fragmenting the matter. In a liquid/liquid system, collapse of the cavitation bubbles near the interface induces disruption and efficient stirring forming emulsions.[6]

The use of combined microwaves and ultrasound devices as a hybrid technology was first broached in the mid 1990s.[7−9] Ultrasound can provide hotspots and strong aforementioned mechanical effects, while heat transfer can be improved with the microwaves' selective heating.[10] The combination of these technologies offers an enthusing mean to focalize two different sources of energy, with their own specific effects. The reduction in energy consumption found in microwave heating, due to short reaction times, and the efficient depolymerization of ultrasound irradiation generate a decrease in reaction time and an increase in yield, leading to overall energy saving.[11] However, this recent technology still displays some limitations, such as small reaction volumes, fragility and insufficient adapted accessories.

This dual technology has been employed successfully in the field of nanotechnology for the synthesis of nanowires,[12] in various organic syntheses,[13−16] in extraction technologies,[17,18] and in biodiesel synthesis.[19] Maeda et al.[7] are known as the inventors of dual microwave and ultrasound irradiation. Chemat et al. developed a combined reactor using a digestion microwave monomode combined with indirect 20 kHz metallic cup-horn probe irradiation used for the enhancement of urea pyrolysis and propanol esterification with acetic acid yields[8] for the digestion and dissolution of biological and chemical products[20] and for the determination of total Kjeldahl nitrogen in a digestion process.[21] The combination of the two technologies increased the mass and heat transfers in the digestion and dissolution processes. The investigation that is most closely related to our work was performed on the hydrolysis of cornstarch into levulinic acid under a combined ultrasound−microwave assisted device by Zhou et al.[22] The levulinic acid yield of 23.17% was achieved in 90 min at 100 °C in 4.5 mol. L^{-1} of hydrochloric acid, with a liquid−solid ratio of 15:1 (mL g^{-1}). Cravotto and Cintras[23] published a short review about the concept of combined irradiation, and the types of reactors available in the literature illustrated with synthesis and catalysis applications. Recently, Ragaini et al.[24] have developed a combined microwave and ultrasound reactor in orthogonal irradiation. The design of the reactor allows processes to be performed in simultaneous and direct irradiation. Microwaves are directly emitted into the bulk (no resonance of microwave electric field) and protected with a glassware tube from the nearby ultrasound emitter. An entire book chapter has been dedicated to combining ultrasound and microwave irradiation in organic chemistry.[25]

The aim of this research was to examine the effect of combined simultaneous microwave and ultrasound irradiation technology on starch-based industrial waste. The starting materials were also irradiated separately with microwaves or ultrasound for comparison with the combined reactor in order to detect any eventual synergetic effect. Synergism can be defined as a phenomenon resulting from the effect of a combination of technologies, tools, or reagents that exceeds the sum of their individual effects.

MATERIALS AND METHODS

Materials. Three different raw materials were utilized for comparison: potato starch, wet potato sludge, and dry potato sludge. Potato starch, employed as a reference due to its composition of amylose and amylopectin, was purchased at the supermarket, while wet and dry potato sludge, which was composed of potato peels, was obtained from the potato industry. The principal components identified in the investigated wet potato sludge are presented in Table 1. Finally, a glucose oxidase/peroxidase assay was purchased from Sigma-Aldrich, while other chemicals were commercially available.

Dual Microwave/Ultrasound Apparatus (MW/US). This combined apparatus incorporates several adaptations to allow double irradiation. Typically, an electric arc can be formed when a metallic horn is placed in a closed microwave cavity, leading to health and safety problems. Subsequently, an ultrasound probe featuring a Pyrex horn (frequency 20.1 kHz with a tip diameter of 17 mm) was introduced inside a professional multimode microwave synthesis (Microsynth, Milestone) (Figure 1). Finally, a two-neck round-bottomed flask was placed into a Teflon reactor and refrigerated at 0 °C with a microwave-inert cooling liquid.

Table 1. Principal Components Identified in Investigated Wet Potato Sludge

principal components of potato	average values obtained in factory source wet potato sludge (%)	average values obtained in potatoes from literature[34](%)
dry matter	19.0[a]	15−28
moisture	81.0[a]	72−88
starch	22.9[b]	12−18
crude protein	7.8[c]	0.6−2.1
crude fiber	3.9[d]	1−2
ash	7.3[e]	
crude fat	0.3[f]	
free sugars	<1	0.2−1.8
phosphorus[g]	180	30−60
nitrogen-free extract	80.7[h]	

[a] Gravimetric determination: sample dried overnight at 105 °C, SFS 3008. [b] Determination of starch content ISO 10520: Polarimetric method. [c] Kjeldahl determination: ISO 1871:1975 and ISO 937:1978. [d] Sample was hydrolyzed with acid and biodegradable fraction with alkali and organic matter, and the end product is the raw fiber: modified standard ISO 5498. [e] Gravimetric determination: sample burnt off after 4 h at 600 °C. [f] Gravimetric determination with ether-soluble portion. [g] Unit is mg 100g^{-1} [h] Soluble carbohydrates calculated by 100% subtracting crude protein, crude fiber, ash, and crude fat.

The acoustic power of the ultrasound Pyrex horn was measured according to the procedure by Kimura et al.[26] An amount of 100 mL of water was irradiated for 240 s, and the temperature was recorded every 20 s. The slope of the resulting linear equation was introduced into the calorific equation; therefore, an acoustic power of 17 W was reached for an electric power of 40 W (20.1 kHz horn), indicating an energy efficiency of 42.5%. Although energy efficiency is an average,[19] the electric power delivered to the Pyrex probe is quite low compared to metallic horn.[3] One possible reason could be the poorer mechanical properties of Pyrex compared to titanium alloys generally used in the metallic horn set up. The utilization of a metallic horn simultaneously in a microwave cavity appeared to be unsafe due to the nature of the horn and the formation of electric arcs. Even so, the prospect of a synergism remains a possibility and an exciting thought.

General Procedures. Experiments were performed in acidic water (3 mol L^{-1} H_2SO_4) with the three types of raw material, as previously stated. A 3 or 13 wt % solution was poured into a 250 mL two-neck round-bottomed flask and placed into the combined device. The solution was irradiated with either ultrasound or microwaves and then simultaneously for 120 min at a temperature of 60 ± 3 °C. The solid phase was separated from the liquid phase by centrifugation for 15 min at 3500 tr min^{-1}. The solid phase was then washed twice with distilled water, centrifuged, and lyophilized for a mass balance calculation. The liquid phase was filtered through a 47 mm nylon filter membrane of 0.45 μm to obtain a clear solution. The solution was neutralized with pellets of sodium hydroxide to reach a basic pH for the total reducing sugar (TRS) analysis.

Analysis. The total reducing sugar content was determined with a 1% dinitrosalicylic acid (DNS) reagent according to the Miller technique.[27] A 2 mL sample of the tested solution was added to 2 mL of the 1% DNS reagent solution and boiled precisely for 5 min. Then, 1 mL of a 40% solution of potassium sodium tartrate was added in order to keep the coloration of the reaction and cooled to room temperature to quench the oxidation reaction. The TRS concentration

Figure 1. Combined ultrasound/microwave system with refrigerated thermostatic bath, developed at the University of Turin.

in each solution was calculated according to a standard curve performed on glucose with an error analysis of 5%. The analysis of the quantity of glucose was executed with the glucose oxidase-peroxidase assay reagent according to the Sigma-Aldrich glucose kit assay procedure. The standardization was carried out on a glucose standard solution provided by the kit. A quantity of 2 mL of assay reagent was added to the solution and heated for exactly 30 min at 37 ± 1 °C. The color of the resulting solution was stabilized with 2 mL of 12N H_2SO_4. The absorbance of each tube was measured against the reagent blank at 575 and 540 nm for the TRS and glucose assay, respectively, with a UV−visible spectrophotometer, named Beckman DU 70. The glucose quantity was not measured when the yield of reducing sugars was below 10%. The morphology and microstructure were characterized by field emission scanning electron microscope (Zeiss Sigma FESEM) at the Centre of Microscopy and Nanotechnology in University of Oulu.

■ RESULTS AND DISCUSSION

Characterization of Raw Materials. Analyses of the average content of the potato sludge (22.9% of starch + 3.9% of crude fiber + <1% of free sugars) corroborated the 29% wet basis obtained from the total hydrolysis (Table 1). In other words, 29% of the wet basis or 88% of the dry basis are sugar potential.

The particle size distribution was performed on the three starting materials, providing information about the different aspects of the particles and the accessibility of the granules for hydrolysis (results in Table 2). Settings of the analyses are displayed in our previous work.[3] Potato starch generated a mode particle diameter of 63 μm. The mode represents the value that occurs most frequently in the distribution. The average size of individual granules differs for each source of starch. The average granular size ranged from 1 to 100 μm, 1 to 20 μm, and 3 to 7 μm for potato starch, maize starch, or rice starch, respectively.[28] The natural materials possessed bimodal distributions at 40 and 1000 μm for the wet potato sludge and at 50 and 450 μm for the dry potato sludge. A mode particle diameter of 50 μm for dry potato sludge showed a high presence of free particles, whereas the one for wet potato sludge reached 1000 μm, which represents more agglomerated particles.

Table 2. Particle Size Distribution of Potato Starch, Wet Potato Sludge and Dry Potato Sludge

material	median[a] (μm)	mode[b] (μm)	population
potato starch	42	63	single (range 10−120 μm)
wet potato sludge	656	1000	dual (40 and 1000 μm)
dry potato sludge	53	50	dual (50 and 450 μm)

[a] Particle diameter that divides the frequency distribution in half. [b] Particle diameter that occurs most frequently.

Microscopic imaging analyses performed pre- and post-hydrolysis revealed the different aspects of the granules (Figure 2). The smooth, ovoid, and large shape of the granule indicates potato starch (Figure 2A). Two hours of sonication eroded the surface of the potato starch granules, although the shape was not altered (Figure 2D). This type of erosion is known as endocorrosion, defined as the formation of pits and holes on the surface of the granule. The surface showed that the intensity of the sonication was not enough to disrupt the granules and release the starch; several granules appeared to be cleaved. These surface alterations have been previously observed in the literature.[29,30] It was also shown that ultrasound degraded preferentially amorphous in the crystalline region. Amylose is favored for degradation due to its linear structure. Microwave irradiation generated a total different result with the apparition of a swelling line on the surface of the potato starch (Figure 2F). It appeared that several granules merged to form single ones without any disruption to the surface. This phenomenon has also been observed previously in the literature[31] with a conventional heating of starch in linoleic acid with α-amylase. The swelling lines breached before they were forming, and the granule opens and releases starch. The combined irradiation of the 3 and 13 wt % potato starch suspension solution degraded the entire starch molecules into water-soluble sugars; no solid phases were recovered. The degradation of amylose produces water-soluble polysaccharides.[10]

Biomass debris can be found surrounding the granules of the two native materials (Figure 2B, C). The granules of the dry material are mainly separated from the debris (Figure 2B), whereas the granules of the wet material are entrapped (Figure 2C). The sonication of the dry potato sludge generated a dark oil residue, and no solid was analyzed. Ultrasonic irradiation of the wet potato sludge disrupted the debris, thereby releasing singular granules to become more accessible for degradation (Figure 2E). Similarities on the surface of some granules were observed with the potato starch (bottom right of Figure 2E compare to Figure 2D). The single microwave irradiation disrupted the entire shape of the granules of the native potato materials (Figure 2G, H), and as a result, no granules were observed because debris was entrapped in the gel structure. This change of structure was solely observed after microwave irradiation of the raw materials, and this too has been observed previously in literature.[32] The combined irradiation of the 3 wt % dry potato sludge suspension disrupted all sizes of granules,

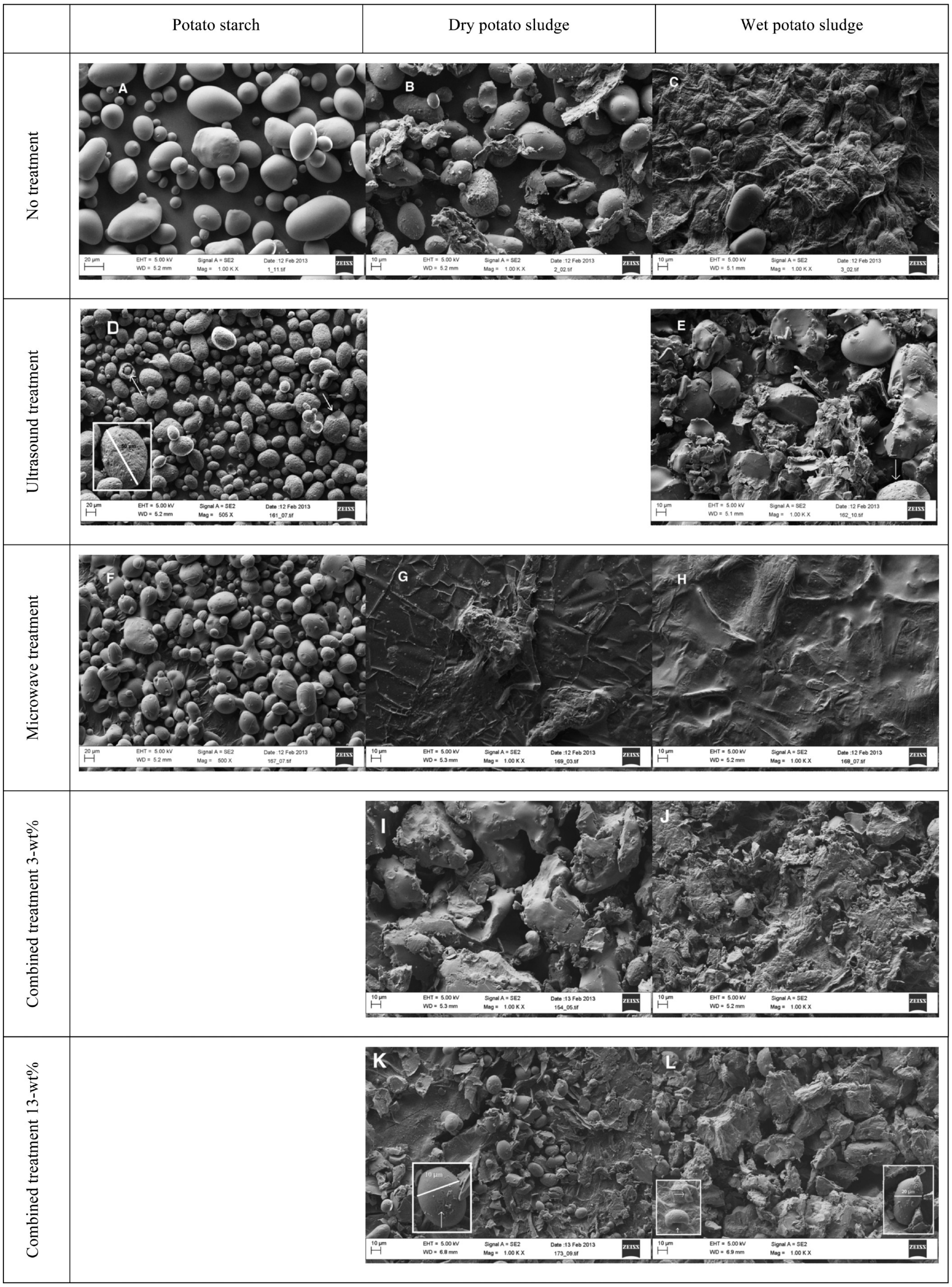

Figure 2. FE-SEM images of different starch materials before treatment: (A) potato starch, (B) dry potato sludge, and (C) wet potato sludge; under various irradiations of a 3 wt % at 60 °C for 120 min in H_2SO_4 3 mol L^{-1}: (D) potato starch after sonication, (E) wet potato sludge after sonication, (F) potato starch after microwave irradiation, (G) dry potato sludge after microwave irradiation, (H) wet potato sludge after microwave irradiation, (I) dry potato sludge after combined irradiation, and (J) wet potato sludge after combined irradiation; and also combined irradiation of a 13 wt % at 60 °C for 120 min in H_2SO_4 3 mol L^{-1}: (K) dry potato sludge and (L) wet potato sludge.

whereas the smaller ones (<10 μm) remained in the 13 wt % suspension solution (Figure 2I, K). The smaller granules are more resistant to acid hydrolysis, which was previously observed in the literature.[33] Certain granules possess erosion attacks on their surfaces (frame of Figure 2K). For example, the combined irradiation of the wet material disrupted the granules,

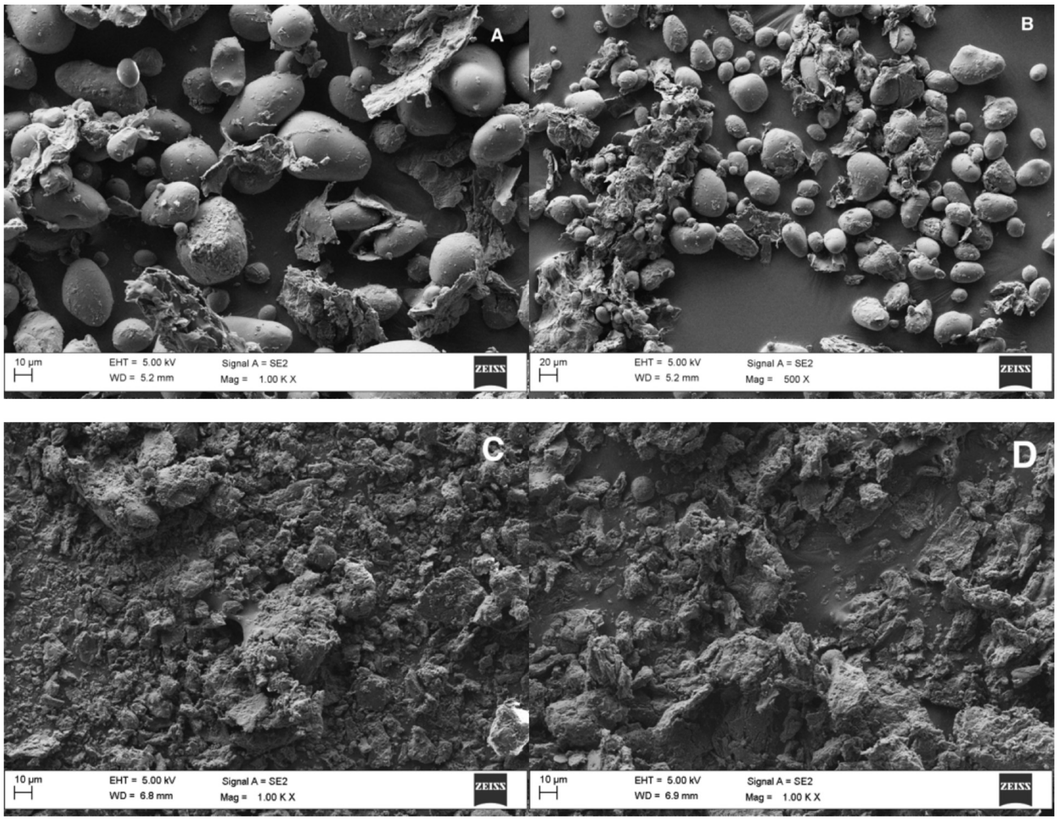

Figure 3. FE-SEM of dry potato sludge before process (A,B) and after 2 h of sonication of 3 wt % suspension in H_2O at 60 °C (C,D).

Table 3. Experimental Conditions and Results of Reducing Sugars and Glucose in Terms of Percentage of Depolymerization of Raw Materials: Potato Starch, Wet Potato Sludge, and Dry Potato Sludge[a]

	experimental conditions			experimental results	
experiments	raw materials	techniques	weight percentage (wt %)	yield of reducing sugars (%)	yield of glucose as the ratio of reducing sugars (%)
1	potato starch	US LF	3.0	18	
2	potato starch	MW	3.0	81	36
3	potato starch	combined	3.0	79	
4	wet potato sludge	US LF	3.0	1	
5	wet potato sludge	MW	3.0	35	21
6	wet potato sludge	combined	3.0	46	
7	dry potato sludge	US LF	3.0	12	
8	dry potato sludge	MW	3.0	87	31
9	dry potato sludge	combined	3.0	57	
10	potato starch	US LF	13.0	76	90
11	potato starch	MW	13.0	71	53
12	potato starch	combined	13.0	77	97
13	wet potato sludge	US LF	13.0	25	20
14	wet potato sludge	MW	13.0	1	
15	wet potato sludge	combined	13.0	12	2
16	dry potato sludge	US LF	13.0	69	59
17	dry potato sludge	MW	13.0	12	
18	dry potato sludge	combined	13.0	57	83

[a] Depolymerization was performed under low frequency ultrasound irradiation (US LF), microwave irradiation (MW), and combined device (combined).

although a few remaining granules underwent surface erosion (3 wt % Figure 2J) or showed the primary signs of gelatinization (13 wt % Figure 2L). The ultrasound released the granules from the biomass debris, which occurred with a single sonication (Figure 2E), while the microwave enhanced the degradation reaction of starch. The particle size distribution corroborated well with the microscopic images (Figure 2B, C); the granules of the dry materials appeared to be more accessible to hydrolysis than the wet potato sludge, which contained more agglomerated particles.

Comparison of Technologies. US- or MW-assisted depolymerization under neutral and basic conditions of each

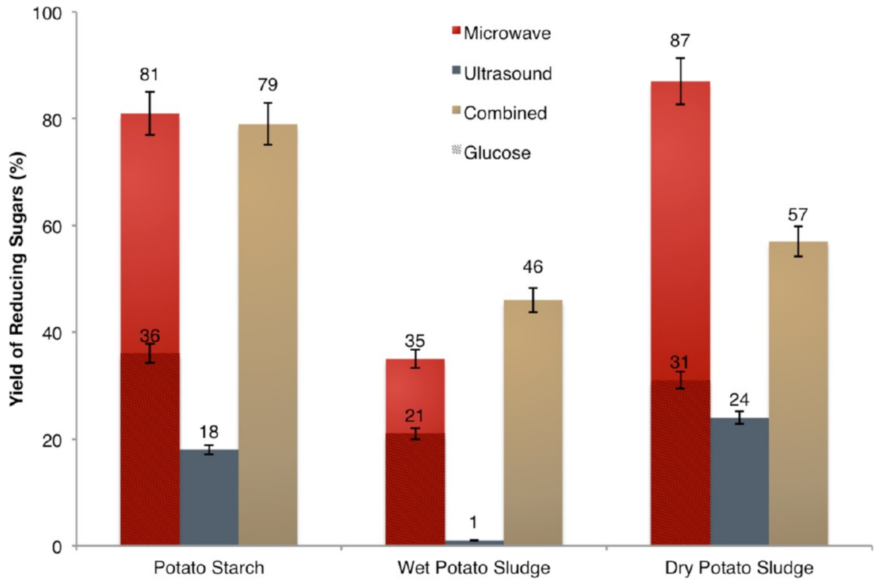

Figure 4. Yield of reducing sugars from the hydrolysis of a 3 wt % suspension solution (potato starch, wet potato sludge, and dry potato sludge), H_2SO_4 3 mol L^{-1}. Depolymerization was performed under microwave irradiation, low frequency ultrasound irradiation, and combined technologies at 60 °C for 120 min. The strips represent the glucose content.

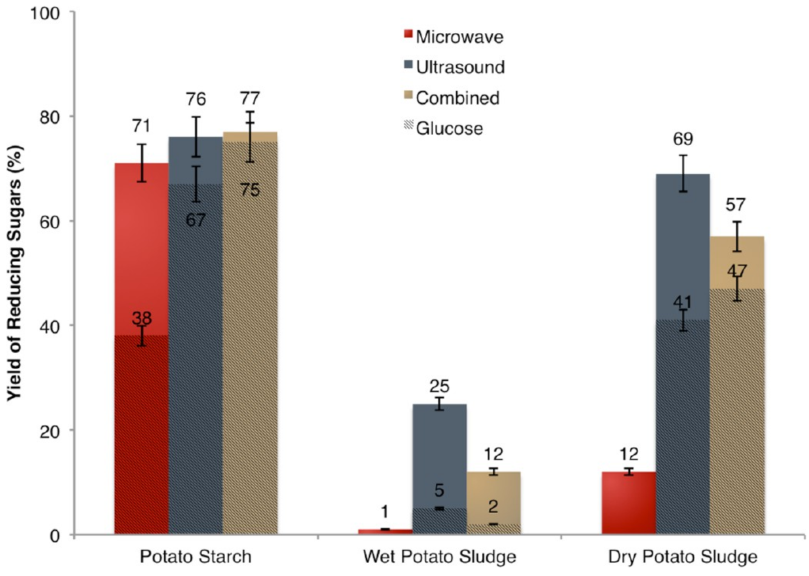

Figure 5. Yield of reducing sugars from the hydrolysis of a 13 wt % suspension solution (potato starch, wet potato sludge, and dry potato sludge), H_2SO_4 3 mol L^{-1}. Depolymerization was performed under microwave irradiation, low frequency ultrasound irradiation, and combined technologies at 60 °C for 120 min. The strips represent the glucose content.

raw starting material was studied in our previous experimental campaign,[3] and no or a negligible quantity of reducing sugars were found. All raw materials irradiated in neutral conditions reached a maximum yield of 5% of reducing sugars (1% for potato starch, 3% for wet potato sludge, and 5% for dry potato sludge). Similar results obtained with single irradiations reflect the natural content of free sugars in the complex matrices. However, a recovered mass balance of the three starting materials showed that 30−40% of the solids were degraded into water-soluble polysaccharides. The long linear and ramified chains of amylose and amylopectin, respectively, can be shortened into water-soluble polysaccharides. Figure 3 represents the sonication of the dry potato sludge under neutral condition (Figure 3C, D) in comparison with the dry material before any processes (Figure 3A, B). The disappearance of the granules suggests that the granule was disrupted and, as a result, released the starch molecules. However, the low yield of reducing sugars suggests that a catalyst is necessary for the degradation of the amylose and amylopectin.

The various technologies were unable to depolymerize the starch molecules, and sulfuric acid 3 mol L^{-1} was therefore added to the process. An increase in the weight percentage was necessary for a potential scaleup of the process; the hydrolysis of 3 and 13 wt % were performed. The experimental conditions and results are summarized in Table 3.

The yields of reducing sugars (TRS) and glucose from the depolymerization reaction of 3 and 13 wt % versus the three types of irradiation are displayed in Figures 4 and 5, respectively. The three raw materials (potato starch and wet and dry potato sludge) were irradiated for 2 h at 60 °C in H_2SO_4 3 mol L^1.

Foremost, the lowest TRS and glucose levels were consistently obtained from the wet potato sludge in all technologies. This complex matrix, containing proteins and minerals, was more resistant to the various types of irradiation. The microscopic images revealed that granules of starch were entrapped in the biomass debris (Figure 2C). The particle size distribution performed in our previous study corroborated the findings that the wet material presented high distribution and therefore agglomerated particles compare to the dry materials. The depolymerization yields of the three raw materials at 3 wt % appeared to exhibit similar behavior. Microwave and combined irradiation generated the highest TRS, whereas ultrasonic irradiation displayed poor results, which seems to tend toward a depolymerization led by a thermal effect. The hydrolysis of 3 wt % potato starch assisted with ultrasound led to 18% of reducing sugars and might be explained by the low efficiency of the Pyrex horn unable to deagglomerate the particles (Table 3, experiment 1). The SEM images (Figure 2D) showed the erosion of the granule's surface but no starch was released. No specific effects were observed with the combined technology. On the contrary, a greater yield of 87% was reached with simple microwave irradiation than with combined irradiation (57%) for the dry potato sludge (Table 3, experiments 8 and 9). The combination of the two technologies reduced the yield of TRS. One possible explanation could be the direct conversion of sugars into levulinic acid by overoxidation. Zhou et al.[22] investigated the hydrolysis of corn meal into levulinic acid, and a yield of 23.17% was reached within 90 min at higher temperature (100 °C) than our experiments (60 °C) and in acidic conditions. Amazingly, the greatest TRS of 46% was obtained with the combined technology with the complex wet potato sludge (Table 3, experiment 6). Indeed, the sonication separated the granules from the biomass debris, while the microwave enhanced the acid hydrolysis (Figure 2J and 2L). On the contrary, the depolymerization did not occur within 2 h of ultrasound irradiation with the single Pyrex probe, probably due to the mechanical weakness of the Pyrex material. Even though the highest yields of reducing sugars were attained with microwave irradiation, the yield of glucose was quiet low, except for the wet material. Moreover, due to the ±5% error analysis of TRS yield, a synergetic effect is hardly conclusive between combined and individual technologies.

Whatever the technology employed, the greatest TRS and glucose yields obtained from the hydrolysis of high molecular weight (13 wt %) demonstrated that microwave, ultrasound, and combined technologies could depolymerize potato starch up to 77% (Figure 5). Significant differences could be observed from the hydrolysis of low molecular weight of 3 wt % (Figure 4). First, the overall view of the graph was inversed, and the ultrasound irradiation generated greater TRS and glucose yields for the natural materials, whereas microwave irradiation was more efficient for low molecular weight (3 wt %). Second, the yield of glucose was twice as high with the combined technologies, where almost 97% and 83% of potato starch and dry potato sludge, respectively, was converted into glucose (Table 3, experiments 12 and 18). Some specific effects may be observed with the combined technologies. In our previous work,[3] a matrix-assisted laser desorption/ionization−time-of-flight (MALDI-TOF) analysis after the process of potato starch (experimental conditions: sonication at 24 kHz for 120 min in sulfuric acid 3 mol L^{-1}) revealed a specific effect of the low frequency sonication. A high amount of short chain oligosaccharides (3 glycosidic units) was noteworthy when compared with other methods of irradiation under identical experimental conditions. The hydrolysis of dry potato sludge with ultrasound and combined irradiations generated 69% and 57% respectively, of reducing sugars. Even though a greater TRS was reached with ultrasound irradiation, the glucose yield was much higher with combined irradiation (Table 3, experiments 16 and 18). The combined irradiation might be glucose selective. The accessibility of the granule and the homogeneity of the matrix allow the irradiation to be more efficient on the potato starch. The lowest yield of reducing sugars and glucose from the hydrolysis of wet potato sludge may be due to the complexity of the matrix with the natural presence of proteins or minerals. The stirring effect provided by the ultrasound required more acoustic power when the weight percentage increased up to 13 wt %. This was confirmed by the SEM images (Figure 2K, L), the smallest granules were more resistant to hydrolysis. A similar observation can be identified for a decrease in the reducing sugars with combined technology, as part of the reducing sugars might be converted into levulinic acid with 2 h of combined irradiation.

At higher weight concentrations, the agglomeration of the particles generated a decrease in the depolymerization rate; the stirring provided by the ultrasound at low frequency might be too weak. In general, the depolymerization rates of reducing sugars and glucose were higher than expected compared with the increase in weight percentage of matter.

CONCLUSION

Combined technology generates a rapid heat transfer of energy into the bulk of the reaction in a significant short time period due to microwave irradiation and an efficient mass transfer thanks to the low frequency ultrasonic irradiation. The two main noticeable results were the high TRS with a natural raw material so far never obtained, especially without any separation processes before the depolymerization, and the glucose selection with combined technology on potato starch. There is a possibility that glucose selectivity may occur with simultaneous combined irradiation because 97% and 83% of the reducing sugars were glucose when the dry materials were irradiated. Because of the high added value of the starch-based industrial waste, its depolymerization into reducing sugars appears to be essential, providing the greatest measured yield of 46%. The combined treatment process related to wet potato sludge indicated a higher yield, compared to individual treatment. However, no significant synergetic effect was observed as expected, except for the wet potato sludge (3 wt %, experiments 4, 5, and 6), although a TRS error analysis of ±5% has to be considered. For most of the treatments, this absence of synergism could come from the main technical hurdle encountered here with a weakness in the mechanical properties of the Pyrex material decreasing the conversion rate of electrical power into acoustical one.

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Notes

The authors declare no competing financial interest.

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