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Hydrothermal Carbonization of Sludge – Optimization for Dewaterability

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***Abstract* – Anaerobic sludge digestate (SD), a by-product from anaerobic digestion, can be used as fuel. However, the high moisture content and poor dewatering properties makes SD unfit for direct energy applications such as combustion and thermal gasification. Hydrothermal carbonization (HTC) process, which uses water as a reaction medium, is a suitable pretreatment method to enhance the dewatering and fuel properties of SD. Hydrochar (HC) obtained after HTC of SD can be used to substitute some fraction of coal for various energy applications. In the present study, HTC of SD was optimized, with respect to dewaterability of HC, using the design of experiment (DoE) approach and the reaction conditions were varied from 158–242 °C and ~10–138 min, respectively. Dewatering assessment of the treated slurry showed that a minimum reaction temperature of 190 °C for 34 min was required to substantially improve digestate's dewaterability.**

***Keywords* – Design of experiment; hydrochar; optimized HTC; sludge digestate; sewage sludge; waste to energy.**

1. INTRODUCTION

Anaerobic digestion (AD) process is widely used in sewage treatment plants (STPs) for sludge stabilisation and additional energy recovery in the form of biogas production. The energy produced during AD of sewage sludge can reduce the overall energy footprint of STPs by 40–75 % [1]. In year 2017 there were around 17 780 biogas plants with installed electric capacity of 10 532 MW across Europe and the number of biogas plants is expected to increase in future [2]. With increasing number of biogas plants across Europe, the amount of sludge digestate (SD – byproduct of AD) is also likely to increase.

During AD, readily degradable organics in feed are converted to biogas, whereas recalcitrant organics are retained in the digestate. Final digestate can have up to 45 % C and 100 % N of the initial feedstock [3]. Conventionally, due to the high nitrogen and phosphorous contents, SD is used in agricultural fields as an alternate source of fertiliser [4], [5]. In addition to land application, digestate has been utilised for various applications such as microalgae cultivation, hydroponics, black solider fly larvae cultivation, fungal cultivation and biogas after some pre-treatment [4]. However, the presence of emerging contaminants, heavy metals, microplastics, and pathogens in digestate poses a severe risk to groundwater as

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well as the quality of soil [6]. In addition, due to strict quality standards by European countries for utilisation of digestate as fertiliser, the amount of digestate that can be used for fertiliser purposes will decrease. Hence, safe, and sustainable disposal of digestate is an emerging challenge for AD operators.

Thermochemical methods such as pyrolysis, combustion and gasification for energy recovery from digestate have been studied extensively in the past [7], [8]. Due to the high moisture content of SD, mechanical dewatering is usually applied before any thermochemical process in order to reduce the energy required by the pre-drying step [9]. The water in SD is normally trapped within highly complex and hydrated polymeric network of extracellular polymeric substances (EPS), making the dewatering operation difficult. It is because, the EPS is produced by microbial biomass, enhancing the floc formation and accounting for 60–80 % of the sludge volume [10]. During conventional dewatering operation, the EPS structure of sludge resists the applied physical forces causing poor dewatering properties. The water content in EPS can vary from 63–99 %, and broadly, the water in sludge EPS can be either in a free state or bound. Free water is the easiest to remove with simple dewatering techniques such as gravity settling and centrifugation. In contrast, eliminating bound water is tedious and energy intensive [11], [12].

In the last decades, hydrothermal (HT) treatments have gained high popularity for management of wet biomass including sewage sludge and SD. HT treatment uses water as reaction medium, eliminating the need of pre-drying the feedstock [13], [14], and facilitates the mechanical dewatering of hydrochar product [3], [15], which becomes more hydrophobic compared to SD. In addition, hydrothermal processes are termed green processes as they do not require any organic solvent or catalysts for the reaction [16] and very little less direct emissions (no secondary pollution). Amongst various HT processes, hydrothermal carbonisation (HTC) has extensively been used by many researchers to enhance the dewatering and fuel properties of wet biomass [17]–[19]. The wet biomass is subjected to a medium temperature (160–240 °C) and self-generated water pressure in this process. Due to the reduction in dielectric properties of water at high temperatures, it behaves like a polar organic solvent [20]. Under these enhanced properties of water, the solubility and reactivity of organics from solid to liquid phase is increased multi-fold. Various chemical reactions such as hydrolysis, decarboxylation, dehydration, condensation, and polymerisation take place parallelly, resulting in formation of hydrophobic hydrochar (HC) as the main product, as well as liquid (process wastewater – PWW), and gaseous by-products.

The fuel properties of HC and PWW are highly dependent on initial feedstock and reaction conditions used during HTC process. Hydrochars produced from HTC process are known for enhanced hydrophobicity/aromaticity, higher caloric value, and improved combustion properties [21], [22]. PWW generated after HTC have high chemical oxygen demand (COD), which could be lowered using membrane processes [23], and various valuable macromolecules such as proteins and humic acids, which can be recovered [24]–[27]. Organics contained in the PWW can be used as a substrate for anaerobic digestion. Previously, HTC has been used on various feedstock such as food waste [28], sewage sludge [29], paper waste [30], digestate [31], [32], waste straw [33], organic fraction of municipal solid waste [34], with the objective of energy densification. However, very few researchers have studied the influence and optimisation of HTC conditions concerning dewatering properties [19], [29]. In the present work, the operating conditions of HTC process were optimised to enhance the dewatering properties of digestate, and HC obtained was further characterised for enhancement in hydrophobicity. Design of experiment (DOE) approach was used to minimise HTC parameters (temperature and time) and maximise the dewatering properties of digestate.

2. MATERIALS AND METHODS

2.1. Feedstock

Dewatered sludge digestate (SD) was obtained from an anaerobic digester located in Wrocław, Poland. The digestate was transported to the lab in a cold container to prevent biological reaction and biogas production. The SD was kept in cold storage (4 °C) to prevent any physio-chemical changes in the waste. Before an experimental run, the sludge sample was shaken vigorously to obtain a homogenised sample. The SD sample was kept in a water bath to bring the sample temperature to room condition.

2.2. Experimental Design

Optimising HTC conditions for dewatering properties was performed using a central composite design (CCD). The effect of two independent variables (time and temperature) at five levels (shown in Table 1) was studied on the improvement in dewatering properties. The upper and lower range of temperature and time were selected based on the published literature [35]–[38]. The design matrix (as shown in Table 1) and statistical analysis of results was performed using Design-Expert (Version 11.0) software. All the runs were performed randomly, and central points were repeated five times to estimate the experimental error.

TABLE 1. INDEPENDENT VARIABLES OPTIMIZED FOR HYDROCHAR DEWATERABILITY

Variables	Symbol	Levels				
		$-\alpha$	−1	0	+1	$+\alpha$
Time (min)	*t*	11	30	75	120	138
Temperature, °C	*T*	158	170	200	230	242

The obtained normalised dewatering values were fitted to second order polynomial regression model Eq. (1):

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k=1} \sum_{i=i+1}^{k} \beta_{ij} X_i X_j \quad (1)$$

where Y is the response (CST), X_i and X_j are independent coded variables. The intercept term, liner, quadratic and interaction effect are represented by β_0, β_i, β_{ii}, and β_{ij}, respectively. Factors and interactions were analysed using analysis of variance (ANOVA) with 95 % confidence level to check model significance. All the insignificant factors were excluded from the final model equation.

2.3. Hydrothermal Carbonization Experiment

HTC runs were performed in a high-pressure reactor (Model number: 4838, Parr Instrument Company). The high-pressure reactor is equipped with a proportional integral controller to maintain the reaction temperature within ±5 °C. For a typical run, ~150 g of digestate (wet basis) was added to the reactor vessel. The reactor vessel was completely sealed and purged with nitrogen gas for 2 min. to create an inert environment. Finally, the reactor vessel was inserted in a heating jacket (pre-heated for ~40 min.) and the reaction time was considered when the reactor achieved the desired set temperature. After the HTC experiment, the reactor vessel was placed under flowing cold tap water to quench the reactions. The slurry obtained after the HTC run was transferred to a container and stored in cold storage until the final analysis.

2.4. Analytical Methods

HTC process influences various physio-chemical properties of sludge, and different standard methods were used to measure the changes in properties of resulting sludge. Total solids (TS) in initial digestate were measured by drying a fixed amount of wet digestate in an oven at 105 °C for 24 h. Total suspended solids (TSS), volatile solids (VS), fixed carbon (FC) and ash content in samples were measured according to the American Public Health Association (APHA) handbook [39] and ASTM standards [40], [41]. The HC yield was obtained by vacuum filtering and drying the sludge cake at 105 °C temperature. The high heating value (HHV) of the dried samples was estimated based on the correlation suggested by Parikh *et al.* [42]. The TS, TSS and mass yield were calculated using Eq. (2), Eq. (3), and Eq. (4).

$$TS\left(\%\right)=\frac{\left(Wt_{\text{initial(wet)}}-Wt_{\text{final(dried)}}\right)}{Wt_{\text{initial(wet)}}}\times 100\,, \tag{2}$$

$$TSS\left(\text{mg/L}\right)=\frac{\left(filter\ paper_{\text{with dried sample}}-filter\ paper_{\text{empty}}\right)}{Sample\left(\text{mL}\right)}\times 1000\,, \tag{3}$$

$$HC\,Yield_{\text{(dry basis)}}\left(\%\right)=\frac{Hydrochar\ weight}{Dry\,SD\ weight}\times 100\,. \tag{4}$$

The severity factor (*f*), which was computed using Eq. (5), was used to analyze the combined impact of reaction temperature and reaction time on different responses.

$$f=50t^{0.5}e^{\frac{-3500}{T}}\,, \tag{5}$$

where *t* and is time (s) and *T* temperature (*K*), respectively. In addition, ultimate composition of the samples was analysed using an Elementar-vario micro cube machine.

2.5. Dewaterability Assessment

Several methods such as centrifugation, time to filter, specific resistance to filter and capillary suction timer (CST) has been reported in literature to measure dewaterability [29], [43]. For the present study, CST was used to estimate the dewatering properties due to better reproducibility of the method. Before CST assessment, the sample was brought to normal room temperature and shaken vigorously to obtain a homogeneous sample. Approximately 5 mL of homogenised sample was transferred to the sample holder in CST instrument (manufacturer: OFI testing equipment). The data obtained during CST measurement was noted down for further analysis. The CST measurements were repeated at least twice, and the data obtained was normalised with respect to the sample's total suspended solids (TSS) as shown in Eq. (6).

$$CST_{\text{normalized}}\left(\text{s}\right)=\frac{CST\left(\text{s}\right)}{TSS\left(\frac{\text{mg}}{\text{L}}\right)} \tag{6}$$

2.6. Hydrophobicity Assessment

A moisture uptake test or hydrophobicity assessment was performed to additionally investigate the influence of HTC reaction severity on the hydrophobic properties of HC product. A more hydrophobic HC will absorb less moisture during storage, transportation and palletisation phase and, therefore, is more desirable [44]. The hydrophobicity of HC sample and initial biomass (dried) was assessed adopting the methodology employed by Bach *et al.* [45]. The biomass and HC samples were grounded and sieved to obtain a fine fraction with a size lower than 250 μm, and the fine fraction was subjected to moisture up-take test. Before moisture up-take test, the samples were pre-dried at 105 °C overnight. Around one gram of dried sample was placed in the climate cabinet (Memmert Climate Chamber HPP1060) and the mass change in the sample was noted for six consecutive days. The climate cabinet was kept at 20 °C temperature and 90 % relative humidity (RH).

3. RESULTS AND DISCUSSION

3.1. HTC Operation with Characterization of Feedstock and HC Product

Characterization results of the SD feedstock and HC product from HTC experiments are presented in Table 2. Initial TS content of the SD was around 12.5 %. Proximate analysis of dried SD revealed ~68 %, ~24 % and ~7 % of volatile, ash and fixed carbon, respectively. Due to high solid content and poor dewaterability of SD, the CST instrument recorded no value for up to 2 hours.

The yield and fuel properties of HC depend on the reaction severity during the HTC process. It can be observed from Table 2 that with an increase in reaction severity, the yield of HC decreased gradually. The highest HC yield of approximately 70 % was achieved for the HTC runs performed at lower reaction temperatures of 158 °C and 170 °C. At the lower reaction temperatures, the yield was observed to be dependent on reaction time. For instance, with an increase in reaction time from 30 to 120 min, an approximately 10 % reduction in HC yield was observed for HTC runs performed at 170 °C. In contrast, the HC yield was observed to be independent of reaction time for the HTC runs performed at a higher temperature of 230 °C. In summary, it can be concluded that the effect of the reaction time on the HC yield becomes less significant for the runs performed at the higher reaction temperatures. The reduction in the mass yield during the HTC process is possibly mainly attributed to the dissolution of organics from solids to the liquid phase and dehydration/decarboxylation reactions.

With an increase in HTC reaction severity, a gradual reduction in volatile fraction was observed in the HC compared to that in the raw SD. Owing to high operation temperature and pressure, organics in solids are solubilised in the liquid fraction, resulting in an overall reduction of volatile matter. The fixed carbon in the HC remained in the 5–10 % range, and no trend with HTC reaction conditions was observed, since HTC also influenced the ash content, by dissolving a part of inorganic fraction. An ash content of approximately 32 % was observed for the HC samples obtained at the lower reaction severities (f = 0.079–0.11). The highest ash content (45 %) was observed in the HTC runs performed at a reaction severity of 0.30. The presence of high ash content in SD inhibits the energy densification process during HTC treatment [46], and only a slight enhancement in the calorific value was observed at the higher reaction severity. The results are in agreement with previously published work [37].

A Van-Krevelen diagram of the HC samples was obtained based on the molar atomic ratios, as shown in Fig. 1.

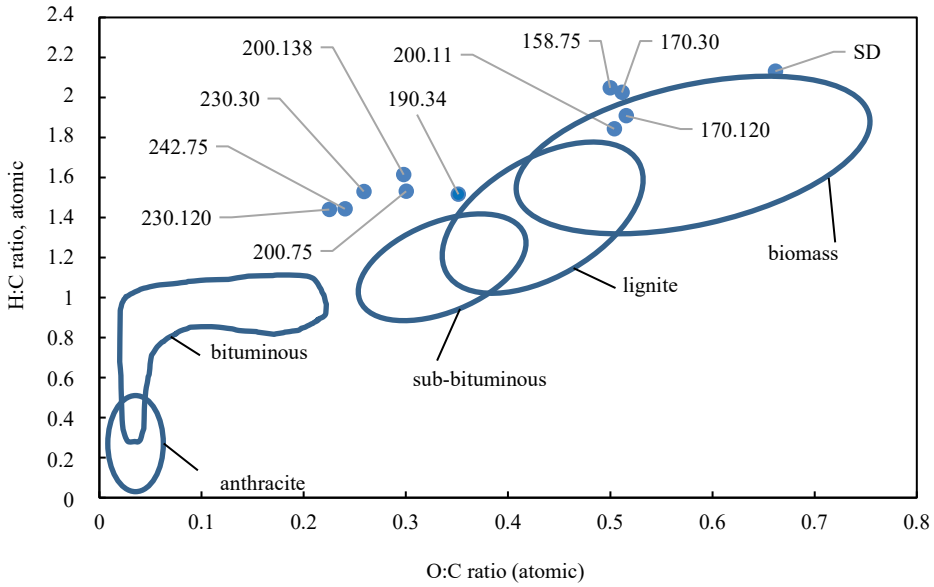

Fig. 1. Van-Krevelen diagram of SD, and HC samples produced at different HTC reaction conditions (caption example 190.34 = 190 °C, 34 min)

TABLE 2. FUEL PROPERTIES OF FEEDSTOCK AND HYDROCHAR SAMPLE OBTAINED AT DIFFERENT REACTION CONDITIONS

Reaction conditions				**Proximate analysis (dry basis)**								**Ultimate analysis**		
T, °C	**Time, min**	**Severity factor, *f***	**Mass yield**	**VM, %**	**FC, %**	**Ash, %**	**C, %**	**H, %**	**N, %**	**S, %**	**O, %**	**HHV (db) (MJ/kg)**	**ED factor**	**Energy yield**
SD	–	–	–	68.17	7.35	24.47	33.5	5.95	6.2	0.33	29.6	14.5	–	–
158	75	0.079	68	59.20	8.55	32.24	33.4	5.7	6.1	0.3	22.3	14.5	1	68
170	30	0.082	70	61.05	5.33	33.60	32	5.4	5.9	0.27	21.82	14.6	1.007	70.5
170	120	0.10	61	59.3	5.74	35.32	32.7	5.2	5.3	0.31	22.47	14.4	0.993	60.6
190	34	0.12	58	50.51	7.8	41.67	33.4	4.22	4.81	0.25	15.7	14.5	1.000	58.0
200	11	0.11	63	58.39	6.98	34.61	33.2	5.1	5.7	0.27	22.3	14.5	1.000	63.0
200	75	0.16	54	51	6.25	42.72	34.4	4.39	4.47	0.25	13.8	15.4	1.062	57.4
200	138	0.18	52	50.5	6.7	42.8	34.2	4.6	4.5	0.32	14.1	15.7	1.083	56.3
230	30	0.21	51	49.8	6	44.12	34.5	4.4	4.8	0.2	11.9	15.8	1.090	55.6
230	120	0.28	47	47.42	6.9	45.61	35	4.2	4.6	0.27	10.5	15.9	1.097	51.5
242	75	0.30	47	44.31	9.95	45.72	35	4.21	3.62	0.24	11	15.8	1.090	51.2

T = temperature; VM = volatile matter; FC = fixed carbon, db = dry basis; ED = energy densification

The HTC of SD resulted in the coalification of biomass, and the degree of coalification was higher for the HTC runs performed at the higher reaction severities. The reduction in H:C and O:C ratios during HTC process is mainly associated with dehydration and decarboxylation reactions [29]. During HTC process, various macromolecules, such as proteins and carbohydrates, undergo polymerisation and reactions, and small molecules of

H_2O and CO_2 are eliminated. The H:C and O:C ratios of the char obtained at a reaction temperature of 170 °C and lower resembled the untreated biomass, that is, the change in their elemental ratio was not substantial. However, for all the HC samples obtained above 190 °C, their elemental properties closely matched those of the lignite and sub-bituminous coals. In contrast, the properties of the HC sample obtained at 200 °C and 11 min reaction duration remained in the biomass region, most likely because of the short duration of the reaction, which prevented the coalification process. The elemental properties of the HC samples obtained under optimum HTC conditions were found to be in the lignite or brown coal region.

3.2. Influence of HTC Conditions on Dewaterability of HC and Optimum Point

During HTC, it was observed that the CST of the treated sample decreased with increasing reaction severity (Fig. 2). The CST and normalised data obtained under the different reaction conditions are listed in Table 3. The dewatering properties of treated SD can be subdivided into three phases: poor, transition, and good dewatering, as shown in Fig. 2. The CST value in the poor dewatering zone resembled that of the original sample, that is, no or minute changes in the dewatering behaviour. In the poor dewatering phase, HTC conditions are not severe enough to cause any substantial damage to EPS structure of the biomass. Moreover, HT treatment of the sludge under 170 °C temperature may have produced fragmented and lower compactness sludge flocs causing the dewatering properties to deteriorate compared to the initial sample [15]. In the transition phase, a slight improvement in the dewatering properties was observed probably due to partial conversion of the bound water to free water. In the good dewatering zone, most of the bound water was converted to free water due to complete elimination of the EPS structure and enhanced the hydrophobic nature of the resulting HC.

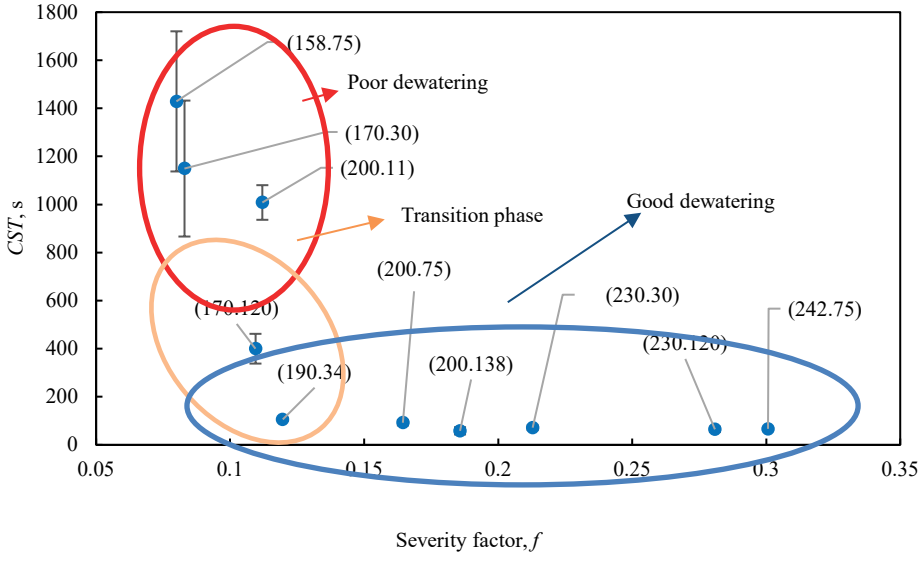

Fig. 2. Influence of reaction severity on CST of SD and resulting HC samples.

TABLE 3. ANOVA TABLE FOR NORMALISED CST RESPONSE

Source	Sum of squares	Degree of freedom	Mean square	F-value	p-value
Model	765.85	5	153.17	35.73	< 0.0001
A-Time	142.26	1	142.26	33.19	< 0.0001
B-Temp	427.37	1	427.37	99.71	< 0.0001
AB	54.86	1	54.86	12.80	0.0027
A^2	27.10	1	27.10	6.32	0.0238
B^2	140.20	1	140.20	32.71	< 0.0001

The highest CST (poor dewaterability) value of approximately 1430 s was observed for the HTC run at the lowest reaction severity of 0.079 (Fig. 2). Under these conditions, a product slurry was obtained without distinct separation of HC and PWW, resulting in higher CST values. With a further increase in the reaction severity, a gradual reduction in the CST value was observed, which could be attributed to the destruction of the EPS structure [47]. Fig. 2 shows that with an increase in the reaction severity from 0.075 to 0.16, the CST value of the treated sample decreased from 1430 to 90 s, respectively. However, only a slight reduction in the CST value was observed with a further increase in HTC reaction severity, which is similar to the results reported by other studies [29]. Interestingly the CST value was 400 s for a severity factor of 0.1 (170 °C for 120 min) and 1000 s (2.5 times) for a severity factor of 0.11 (200 °C for 11 min). Hence, reaction time is also significantly influencing the dewatering properties of digestate, especially at the lower reaction temperatures. This suggests that a run performed at a higher temperature but for an inadequate reaction duration may not enhance the digestate's dewatering. Due to complete destruction of the EPS structure, the influence of HTC reaction time on dewatering becomes insignificant beyond a specific threshold temperature [15].

The normalised CST data was statistically analysed using analysis of variance (ANOVA) with 95 % confidence level to identify the significant input parameters. ANOVA (Table 2) showed that all the input parameters i.e., time, temperature, and their interactions, were significant (i.e., p value less than 0.05). Hence, for the final model equation, all the parameters were considered. Moreover, the values of R^2 (0.92) and predicted R^2 (0.83) were above 0.8; therefore, the model could be used for prediction [47]. The contour plot for normalised CST (Fig. 3) revealed that beyond 200 °C, there was no substantial improvement in the dewatering properties. However, at the lower HTC temperatures, the dewatering properties were found to be time dependent. Kim *et al*. (2014) [29] also reported a similar trend in dewaterability property after HTC of SD.

Actual and coded equations obtained for the response variable are represented as Eq. (7) and Eq. (8). The optimisation of HTC conditions was carried out to achieve enhanced dewatering properties i.e., minimising the CST value with least severity. Based on the model equation, the optimal reaction temperature and time were 190 °C and 34 min, respectively. Under optimum HTC conditions, a CST value of ~100 s was recorded for the treated slurry.

$$CST_{\text{normalised}} = 241.52 - 0.57 \times t - 1.99 \times T + 0.0019\, t \times T + 0.0008 \times t^2 + 0.0041 \times T^2, \quad (7)$$

$$CST_{\text{normalised}} = 0.92 - 2.98 \times t - 5.17 \times T + 2.62\, t \times T + 1.62 \times t^2 + 3.69 \times T^2, \quad (8)$$

where T is HTC temperature in °C, and t is the residence time in minutes.

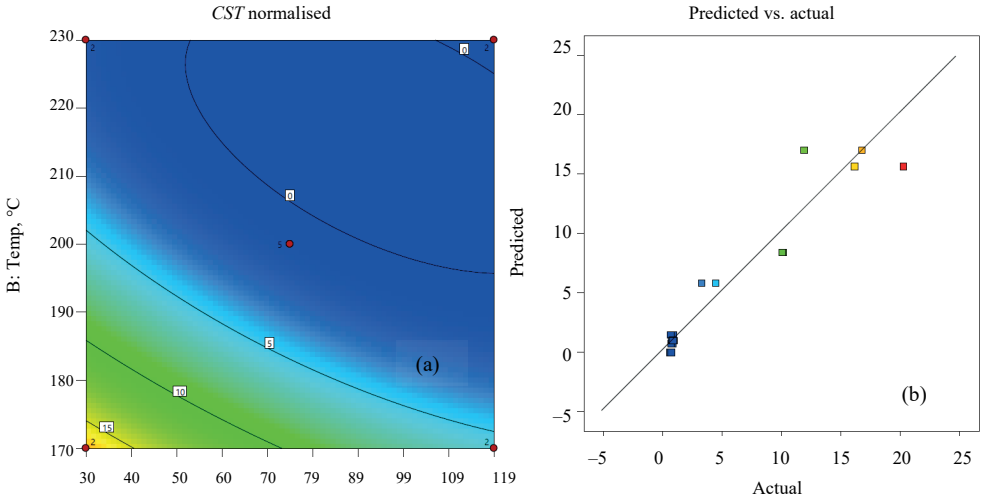

Fig. 3. (a) Contour plots for CST change with temperature and time; (b) fit of predicted and experimental value.

In summary, during HTC process, the dewatering properties of SD are enhanced via two pathways: (i) destruction of EPS structure and (ii) enhanced hydrophobicity of the solids present in SD. It is because, as discussed earlier, the water in the SD exists in two forms: free water and bound water [12]. The first form, free water, is easy to separate using standard methods such as centrifugation, thickening, and settling. However, bound water is tightly bound within the complex structure of digestate biomass, extracellular polymeric substances (EPS), and resists mechanical dewatering methods [46]. Because of the high temperature and pressure, the HTC process damages the highly hydrated EPS structure, resulting in the transformation of bound water to free water and improved dewaterability. Moreover, HTC process transforms biomass's chemical and functional groups composition, making them more hydrophobic in nature [48].

3.3. Hydrophobicity Assessment

As discussed in section 3.2, two major factors that influence the dewatering properties are: (i) EPS destruction and (ii) hydrophobicity of solids (HC). A moisture uptake assessment was performed to further examine the influence of HTC on the hydrophobic properties of HC, the result is shown in Fig. 4. It was observed that untreated samples i.e., SD reached a plateau of the moisture uptake of approximately 70 %, for RH 90 % and temperature of 20 °C, within the first 24 h of assessment, and their respective weights remained constant for the next six days. The HC obtained at least severe HTC conditions (150 °C and 75 min) showed a slight enhancement in hydrophobic properties and a gradual increase in moisture uptake was observed during the assessment, further confirming the hypothesis that lower reaction temperatures are not suitable for achieving better hydrophobicity.

In contrast, HC obtained at the highest reaction severity (242 °C and 75 min) showed the lowest moister uptake due to higher hydrophobic nature. This further confirms the influence of reaction severity on the properties of HC. A maximum moisture uptake of around 35 % was observed for the HC obtained at optimum HTC condition. In summary, the hydrophobic properties of HC were found to be dependent on the reaction severity.

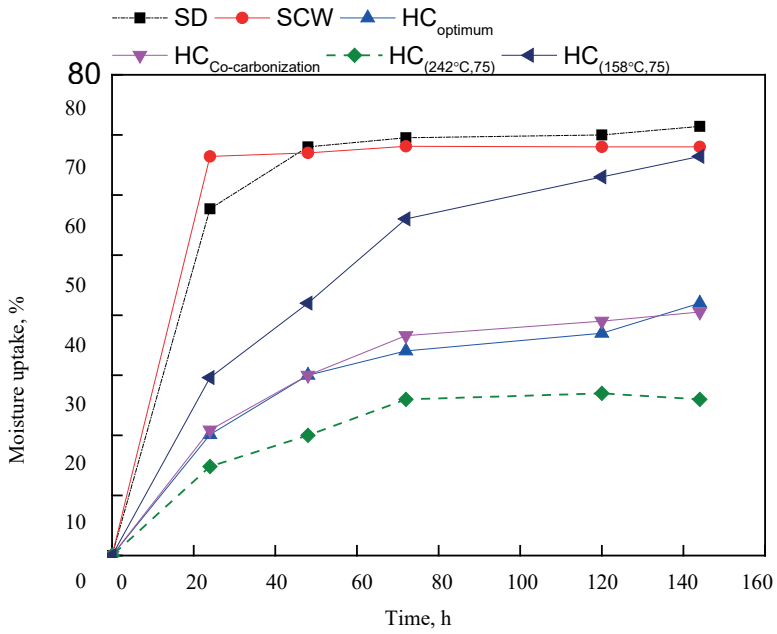

Fig. 4. Influence of HTC on hydrophobicity of HC (SCW and $HC_{co\text{-}carbonization}$ are not included in this paper).

4. CONCLUSION

HTC process is an efficient method to enhance the overall dewatering and thermal properties of SD. In the present study, an optimum reaction condition of 190 °C for 34 min, with respect to improving the dewatering for SD, was determined. Both parameters: temperature and time, play a significant role during HTC process. However, the significance of time tends to diminish at higher reaction temperatures. At the optimum conditions, the hydrophobicity of the resulting HC was 30 % more compared to raw SD. Moreover, increasing the HTC reaction severity beyond the optimum conditions did not result in a substantial gain in energy densification. Coalification of the SD seems to be beneficial in terms of the use of hydrochars as a solid fuel. Future studies should include the influence of mixing SD with other feedstock on dewatering and energy densification properties of resulting HC.

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