



Thermal Disposal of Post-processing Water Derived from the Hydrothermal Carbonization Process of Sewage Sludge

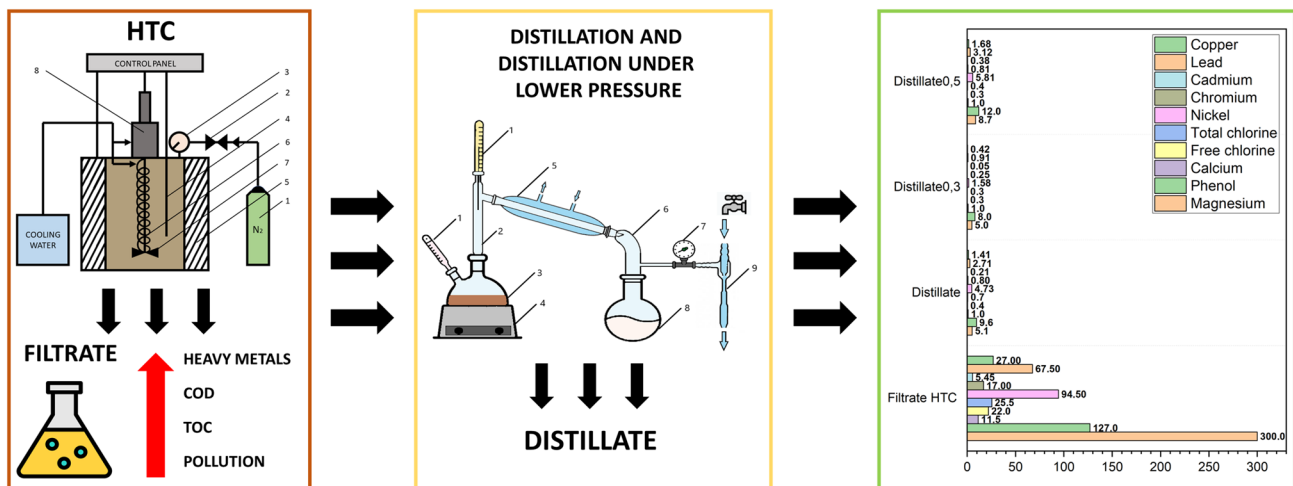
Klaudia Czerwińska¹ · Maciej Śliz¹ · Małgorzata Wilk¹

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Abstract

Hydrothermal carbonization is a suitable method for sewage sludge management due to great improvements in its dewaterability and the successful application of its solid product, hydrochar, in different sectors. However, the resulting liquid product requires special treatment due to the amount of undesirable compounds it contains. The main purpose of this work is to study the use of distillation as a method of post-processing liquid purification. In addition, the influence of vacuum depth on the properties of liquid products was investigated. A number of chemical and physical parameters were determined to confirm the validity of this treatment method: TOC, COD, total nitrogen Kjeldahl content, ammonium nitrogen content, phosphorus, magnesium, calcium, selected heavy metals content and phenol index. Distillation under atmospheric pressure caused reductions in the following parameters: COD, TOC, phenol index, heavy metals, chlorine by more than 90%: removal of 95% COD and TOC, 99.5% of PO₄-P, 93% of Phenol, over 90% of heavy metals, and over 97% of free and total chlorine. In the case of distillation under -0.3 bar pressure, the following reductions were obtained: 97% of COD, 98% of TOC, 99.9% of PO₄-P, 94% of Phenol, c.a. 98% of heavy metals, and more than 98% of free and total chlorine. Decreasing the pressure to -0.5 bar did not produce any significant effects as the results were similar to distillation under atmospheric pressure. Both methods, distillation and distillation under lower pressures, can be used as an appropriate method of post-processing water utilization.

Graphical Abstract



Keywords Hydrothermal carbonization · Sewage sludge · Post-processing water · Vacuum depth · Thermal conversion · Distillation

Statement of Novelty

The aim of this study is to investigate the effective management of highly contaminated liquid derived from the hydrothermal carbonization process of sewage sludge and to optimize the lower pressure distillation process.

Introduction

The constantly growing number of sewage treatment plants necessitates the demand for proper management. The treatment of wastewater results in significant amounts of sewage sludge, which is a biodegradable waste product with a heterogeneous composition that makes safe treatment difficult. In addition to nutrients and organic components, the sludge contains many domestic and industrial impurities such as pharmaceutical residues, heavy metals, hormones, pathogens or microplastics [1, 2].

In the European Union (EU), sewage sludge is disposed of in a variety of ways: through agriculture, storage, composting, incineration and other uses. In 2020, 9.6% of the sludge was placed in storage, 22.4% was used for agriculture, 11.7% composting and other applications, and 35.1% incinerated. The remaining quantity was classified as “other” [3] (Fig. 1). A focus on environmental protection and changes in EU legislation in this area has provided a law which emphasizes the need for education concerning the amount of sewage sludge in landfill [4, 5].

Municipal sewage sludge is increasingly used in agriculture. However, organic and inorganic contaminants contained in sewage sludge can be harmful to the ecosystem [6]. Therefore, an additional element of sewage sludge treatment is required to eliminate any hazards. One of the recently discussed thermal methods for treating sewage sludge is hydrothermal carbonization (HTC). Hydrothermal carbonization

is a thermochemical process in which heat and pressure are used to convert raw biomass and organic waste. This process requires the presence of water, which means that the feedstock does not need to be dried [7, 8]. The energy required for the HTC process is significantly lower when compared to pyrolysis or gasification. The products of this process are hydrochar, liquid and gaseous phases. HTC reduces the volume of waste and enables the recovery of valuable components. In addition, the process improves the dewatering of waste as well as the fuel properties of solid products [9, 10].

Hydrothermal carbonization produces a post-processing liquid, which is the most important by-product as the process causes most inorganic substances to enter the processing water. A large quantity of inorganic compounds, i.e. aldehyde, phenol, alkene, furan and others, have been detected in this liquid. The liquid also contains organic compounds and short-chain organic acids such as benzoacetic acid, butanoic acid, and propionic acid [7, 11, 12]. It has also been found that large amounts of nitrogen (40–70%), phosphorus (50–70%), and potassium [13] can be dissolved in the liquid. Smith et al. [14] showed high concentrations of chlorine, fluorine and bromine, while magnesium and calcium cations were determined at lower concentrations. Mihajlović et al. [15] described two-, three- and four-cyclic PAHs. Luoren, fluoranthene, phenanthrene and pyrene have also been detected in quantities exceeding the permitted levels for drinking water. Xiong et al. [13] found that most heavy metals exceeded acceptable limits, where the amount depended mainly on the reaction temperature. Unfortunately, a large quantity of these substances in the liquid phase require a transformation of the liquid phase (Fig. 2).

In recent years, the transformation of processing liquid by aerobic oxidation, anaerobic fermentation and wet oxidation has been considered.

Langone et al. [16] described the aerobic biodegradability of the process water produced by the hydrothermal carbonization of dewatered anaerobically digested sludge.

Fig. 1 Management of sewage sludge in the EU, 2020 [3]

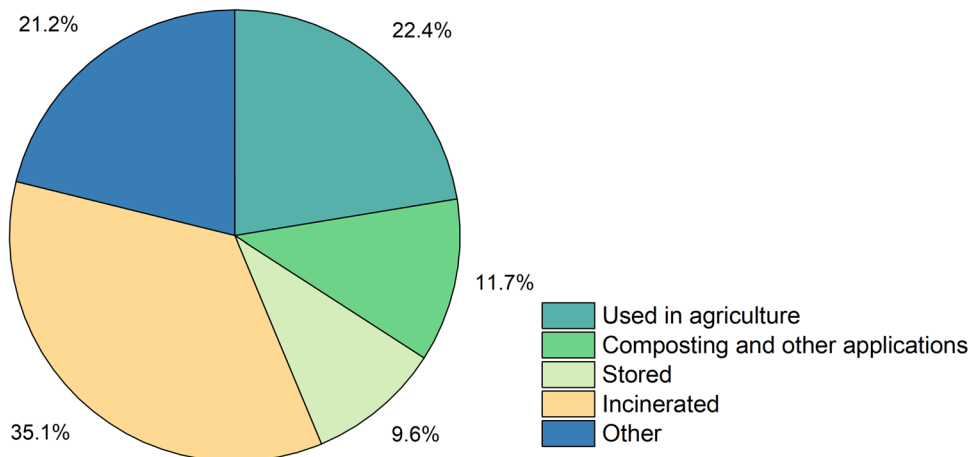
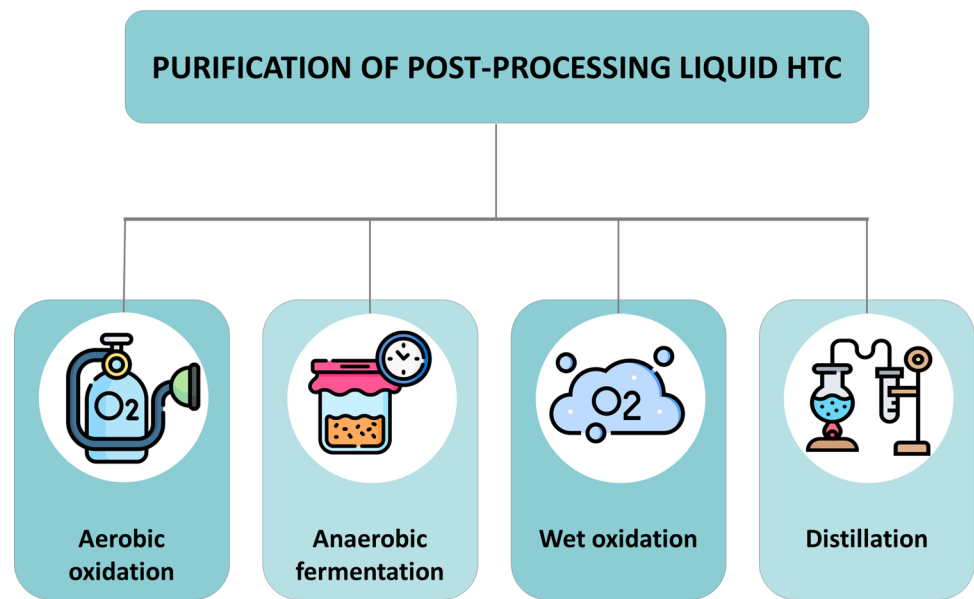


Fig. 2 Purification of HTC post-processing liquid

The liquid proved to be highly biodegradable, up to 83% of the total Chemical Oxygen Demand (COD). In addition, it contained a high concentration of volatile fatty acids, of which the major constituent was acetic acid. Weide et al. [17] also investigated the aerobic oxidation of liquid produced by hydrothermal carbonization and had consistent observations. Anaerobic fermentation was studied by Gaur et al. [18] with the aim of quantifying energy recovery from hydrothermal products (hydrochars and processing water) as solid fuels and raw materials for anaerobic fermentation (AD). The biomethane yield was found to depend on the intensity of the HTC response. A higher biomethane yield was achieved for process water with a lower reaction intensity. Chen et al. [19] used anaerobic digestion to treat the post-processing water from the HTC of sewage sludge to produce CH_4 . Optimal results were achieved when the HTC of SS was performed at 170 °C resulting in a methane yield of 286 mL CH_4 /g chemical oxygen demand (COD). Moreover, the residual liquid phase after the anaerobic fermentation process requires further treatment because anaerobic fermentation does not convert all organic components.

Wet oxidation of the HTC liquid was processed by Weiner et al. [20] as well as Thomsen et al. [21]. Weiner et al. noted that wet oxidation resulted in a satisfactory removal of the chemical oxygen demand and the dissolved organic carbon. Oxidized water proved to be a good substrate for subsequent anaerobic fermentation. Thomsen et al. also observed the positive effect of wet oxidation on the treatment of liquid HTC. The same conclusion was confirmed by Wilk et al. [22]. Both Stutzenstein et al. and Reza et al., as a result of their research, found that wet oxidation was a promising technology to treat the liquid phase produced from the HTC of sewage sludge [23, 24]. The advantage of this process is

that it can reduce the value of TOC and decrease the content of low chain organic acids in the liquid phase. However, this is not a good approach because of its strict experimental conditions [25].

Additionally, there are alternative methods used for the purification of liquids such as distillation, membrane filtration, coagulation or ozonation. The water after the HTC process was previously purified using membrane filtration by Urbanowska et al. [26]. The ultrafiltration process reduced chemical oxygen demand to 30%, biological oxygen demand to 10% and dissolved organic carbon to 21%.

The distillation process was performed by Wilk et al. [27], who distilled the liquid after the hydrothermal carbonization of the sewage sludge and found that this process significantly reduced parameters such as COD and conductivity. The disposal of liquid from the hydrothermal carbonization of biomass was also investigated by Wilk et al. [28], who purified the HTC post-processing water derived from *Miscanthus Giganteus* and acacia. The same team distilled the liquid from the HTC of pine, *Virginia Mallow* and straw receiving very promising results [29]. These studies confirmed that the distillation processes were adequate for liquid treatment after the hydrothermal carbonization of different feedstocks. Distillation takes advantage of differences in the volatility of the components of a mixture at a given temperature. During distillation, the liquid is heated and the resulting vapours are discharged into a condenser from which, after condensation, they are directed to one or more receiving vessels.

The aim of this study is to determine the parameters of the post-processing liquid and to present its distillation. The novelty of this work, compared to previous papers [22, 27–29], is the preliminary study of the influence of the vacuum depth of distillation on the properties of liquid

products. Chemical and physical indicators were determined to confirm the validity of this treatment method and to select the optimal distillation conditions.

Material and Methods

Material

Digested sewage sludge was collected from the Wastewater Treatment Plant in Żory (Poland), then stored at 4 °C to avoid or slow down the biodegradable process. In order to ascertain that the physical and chemical properties of raw sewage sludge did not change, the time of storage was limited to one week. The raw sewage sludge contained 83% moisture.

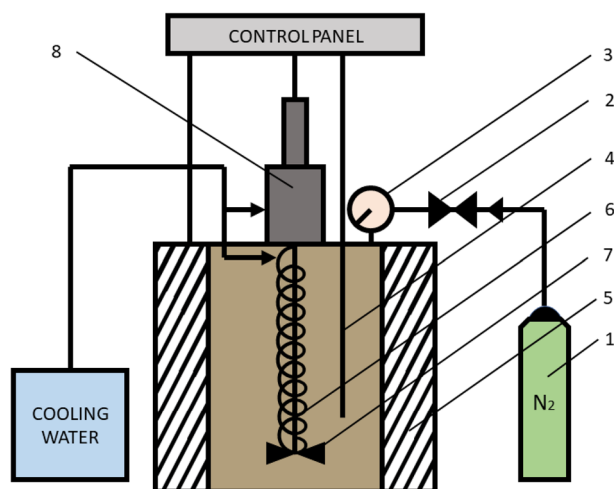


Fig. 3 The HTC set-up (1—Nitrogen cylinder, 2—valve, 3—manometer, 4—thermocouple, 5— electrical furnace, 6—cooling coil, 7—stirrer, 8—magnedrive)

Hydrothermal Carbonization Procedure

Prior to the hydrothermal carbonization test, the raw material was diluted with distilled water in sufficient quantities to ensure easy mixing. The solution was then placed in a reactor chamber, namely a ZipperclaveSteelReactor with a capacity of 1000 ml plus a MagneDrive agitator (Parker Autoclave Engineers). The reactor is described in detail by Wilk [30]. A schematic of the HTC apparatus is shown in Fig. 3.

The reactor was heated up to 200 °C, and when this temperature was reached, the solution was maintained therein for 2 h. Then, the reactor was cooled down via a cooling coil by cooling water. The solution was evacuated from the reactor and separated by a filtration apparatus. Filtration was carried out using a Buchner funnel and a filter paper (qualitative, soft filter, 84 g/m²; type 388 (Filtrak, Germany)). The liquid was placed in an airtight container for further analyses and processes. The HTC procedure is presented in Fig. 4.

Distillation

Distillation is an operation where a liquid mixture is separated into its components. It involves vapourizing the liquid, then collecting and condensing the resulting vapours. The liquefied vapour, which is richer in the volatile component, is called distillate, while the unvapourized residue is called exhausted liquid. The basis of separation here is the difference in volatility of the components. Three distillation processes were conducted at different manometric pressures: atmospheric pressure, -0.3 bar and -0.5 bar. A schematic of the distillation apparatus is shown in Fig. 5. The distillation parameters are presented in Table 1.

Analyses of Filtrate and Distillate

HTC filtrate, called filtrate, and distillate were analyzed to evaluate the treatment effect and influence of the vacuum

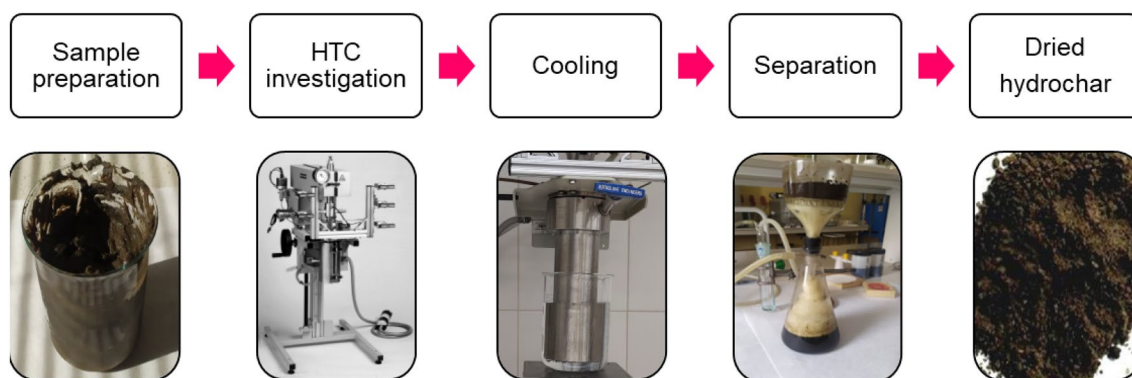


Fig. 4 The HTC procedure

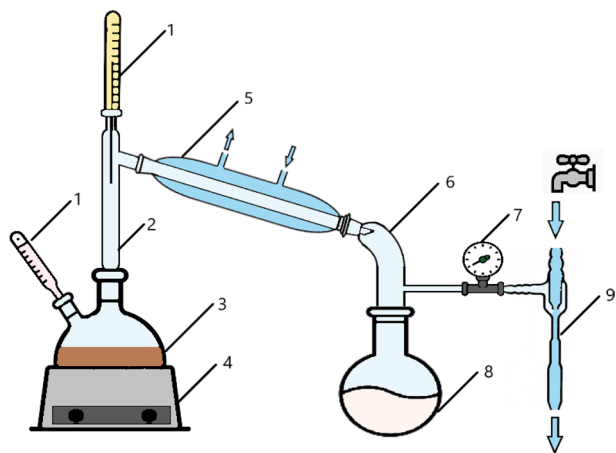


Fig. 5 The distillation set-up (1 – thermometer, 2—still head, 3—round bottomed flask, 4—heat source, 5 – condenser, 6—still receiver, 7 – manometer, 8—receiving flask, 9 – water pump)

depth in distillation on liquid parameters. The following tests were performed: pH, conductivity, chemical oxygen demand (COD), concentration of NH₄-N and PO₄-P, total organic carbon (TOC), Phenol test, Mg, Cu, Ni, Ca, Cr, Cl₂, Cd, and Pb content.

The pH and conductivity were monitored by the multifunctional analyser CX-461 (Elmetron, Zabrze, Poland).

COD, concentration of NH₄-N and PO₄-P, total organic carbon (TOC), Mg, Cu, Phenol, Ni, Ca, Cr, Cl₂, Cd, and Pb content were determined using a Spectrophotometer Merck Spectroquant Prove 100. The standard deviation of the measurement is: COD: ±29.2 mg/l COD; NH₄-N: 0.9 mg/l NH₄-N; PO₄-P: ±0.026 mg/l PO₄-P; TOC: ±9.5 mg/l TOC; Phenol: ±0.019 mg/l Phenol; Mg: ±0.64 mg/l Mg; Cu: ±0.044 mg/l Cu; Ni: ±0.027 mg/l Ni; Ca: ±0.2 mg/l Ca;

Cr: ±0.015 mg/l Cr; Cl₂: ±0.036 mg/l Cl₂; Cd: ±0.0038 mg/l Cd; Pb: ±0.021 mg/l Pb. When heating of the sample was required, the Thermoreactor Merck Spectroquant® Series TR 420 was employed. Appropriate tests from the manufacturer Merck were used to measure these parameters according to the instructions included in the package. In order to determine the total content of the elements Mg, Cu, Ni, Ca, Cr, Cd, and Pb, the samples were mineralized with a special crack set from Merck prior to the appropriate tests.

Results

Characteristics of Filtrate After Hydrothermal Carbonization

The distillation processes showed differences in the characteristics of the filtrate and distillates. There were clear changes in the colour of the individual liquids, as can be seen in Fig. 6. The darkest liquid was obtained for filtrate from the hydrothermal carbonization process. Whereas a lighter coloured liquid was obtained after distillation under atmospheric pressure, while the brightest and similar in shade were obtained after distillation at -0.3 bar and -0.5 bar. A lighter colour may indicate a lower organic content in the liquid [31].

Filtrate, after the hydrothermal carbonization process, is characterized by a high concentration of organic matter, as indicated by the values of TOC, COD, the content PO₄-P and NH₄-N (Table 2). Both COD and TOC levels are very high. The filtrate has a COD of 46 210 mg/L and a TOC of 16,900 mg/L. Langone et al. [11] confirmed that both TOC and COD concentrations in HTC processing waters from sewage sludge can vary in a wide range from 4,000 mg/L

Table 1 Distillation parameters

	Initial mass [kg]	Manometric pressure [bar]	Boiling point [K]	Mass of distillate [kg]	Mass of distillation residue [kg]
1	0.3	0	373.15	0.279	0.021
2	0.3	-0.3	361.15	0.213	0.087
3	0.3	-0.5	350.95	0.177	0.123

Fig. 6 Filtrate (a) and distillates (b) under atmospheric pressure, (c) -0.3 bar and (d) -0.5 bar

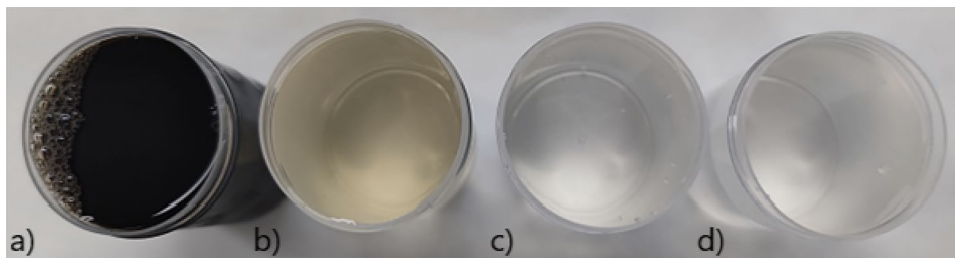


Table 2 Characteristics of the filtrate after hydrothermal carbonization and liquids derived from distillation performed at – 0.3 and – 0.5 bar

	Filtrate	Distillate	Distillate_0.3	Distillate_0.5	Unit
Chemical Oxygen Demand (COD)	46,210	2,600	1,180	1,895	mg/L
Ammonium Nitrogen (NH ₄ -N)	655	950.0	550.0	1,000.0	mg/L
Phosphate (PO ₄ -P)	2,585.0	9.8	4.3	5.5	mg/L
Total Organic Carbon (TOC)	16,900	796	344	534	mg/L
pH	7.05	9.43	9.91	9.96	–
Conductivity	10.880	2.110	0.502	0.992	mS/cm

to 24,000 mg/L, and from 10,000 mg/L to 64,000 mg/L. The results for distillates suggest that distillation may be a suitable method for post-processing liquid treatment, since the distillation process leads to a reduction of COD parameters. Furthermore, distillation under atmospheric pressure reduced COD by less than 95%. During distillation, with a pressure of – 0.3 bar, the COD value was reduced by almost 98%. At a lower pressure of – 0.5 bar, this parameter was reduced by 96%. Wilk et al. [32] also obtained a reduction of the COD parameter when using the distillation process. A greater reduction of the COD parameter during distillation at reduced pressure may be due to the fact that a smaller amount of organic compounds was transported together with the steam to the distillate. This is probably related to the lower distillation temperature during distillation at reduced pressure. The TOC parameter also significantly decreased. Regarding TOC, the greatest reduction was achieved with distillation at – 0.3 bar. For atmospheric pressure, a reduction of around 95% was achieved, in the case of – 0.3 bar, approximately 98%, whereas with – 0.5 bar about 97%. Additionally, for this case, a greater reduction in the TOC parameter during distillation at reduced pressure may be associated with a lower distillation temperature.

A relatively high content of NH₄-N and PO₄-P was also noticed in the filtrate. The content of NH₄-N was 655 mg/L, while the content of PO₄-P was 2,585 mg/L. He et al. [33] found that the HTC treatment has an effect on the release of nitrogen compounds into the process waters. In their research, almost 60% was released into the liquid phase. HTC temperature can strongly influence the N decomposition in HTC by-products. The literature reports that HTC essentially stabilizes phosphorus during the processes as P is mainly immobilized and retained in the hydrochars [34]. Other conclusions were drawn by Aragón-Briceño et al. [35], who reported that phosphorus occurs mainly in process waters in the form of inorganic P. Varying outcomes of phosphorus during HTC depends on the process conditions, type of raw material and content of elements such as Al, Ca, Fe and Mg in the raw material [34].

The NH₄-N concentration increased with distillation under atmospheric pressure, which was less than 32% higher compared to the HTC filtrate. When distillation at -0.3 bar was applied, this value was reduced by 16%

compared to the HTC filtrate. Whereas, distillation at -0.5 bar caused an increase of 34%. Organic nitrogen is the main source of ammonium nitrogen in wastewater. The increase in ammonium nitrogen may be caused by the conversion of inorganic nitrogen into ammonium nitrogen [36]. Another reason for an increase in the ammonium nitrogen content of the sample is the volume of the resulting distillate. The boiling point of ammonium is lower than the boiling point of water. For distillation at a lower pressure of -0.5 bar, this volume was the smallest of all samples. It is likely that the same amount of ammonium was distilled with a smaller amount of distilled liquid. For this reason, the ammonium concentration could have been higher.

The PO₄-P content was reduced by distillation in all cases by about 99.5%. For distillation under atmospheric pressure the value was 9.8 mg/L, at -0.3 bar it was 4.3 mg/L and at -0.5 bar it was 5.5 mg/L. Such a high reduction in phosphate content is probably due to the fact that phosphorus, unlike nitrogen, is not volatile and does not form any stable gaseous compounds [37]. Additionally, the magnesium and calcium content was greatly reduced (Fig. 7). The large reduction of these elements is probably because these compounds are non-volatile. The content of free and total chlorine was also determined in the filtrates and distillates (Fig. 7). In both cases, a drop in content was observed after distillation. In all cases, the decrease was between 97 and 99%. This is probably because the organic content in filtrate is of an amount which can easily go through the distillation process, while some part requires much deeper pressure to distill and remain in the residual liquid.

In addition, the pH and conductivity of the HTC filtrate were determined (Fig. 8). The filtrate was alkaline and had high conductivity—10.88 mS/cm. Considering the conductivity value, which depends on the electrolyte value or its decomposition into ions in liquid, it can be assumed that the HTC post-processing liquid contained a high concentration of electrolytes. There was a noticeable depreciation in conductivity in the distillate. The conductivity of distillates at atmospheric pressure was 2.11 mS/cm, for pressure at – 0.3 bar it was 0.502 mS/cm and for pressure at – 0.5 bar it was 0.992 mS/cm. This indicated that there were some polar compounds in the solution.

Fig. 7 Magnesium, phenol, calcium, total and free chlorine contents in filtrate and distillates [mg/L]

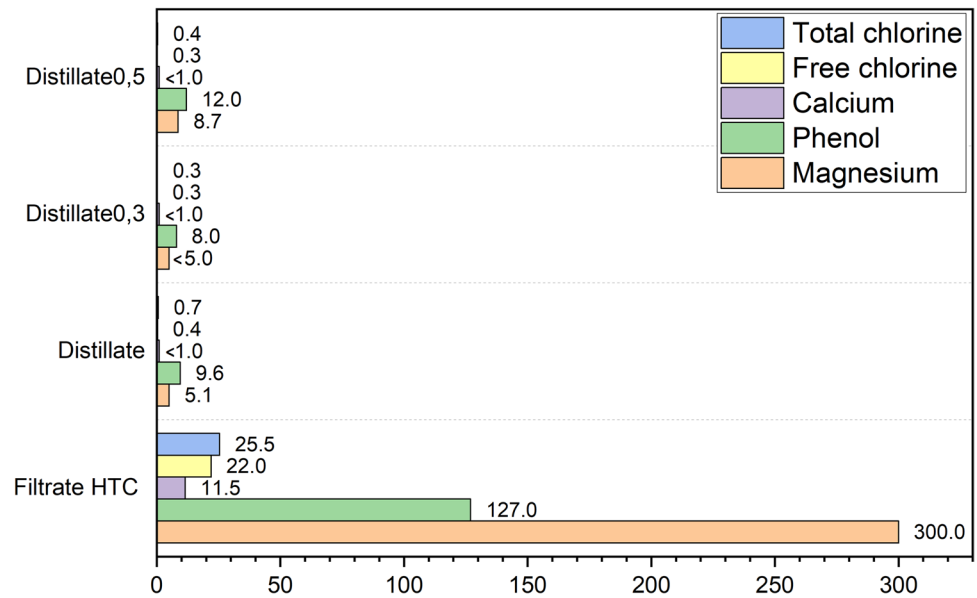
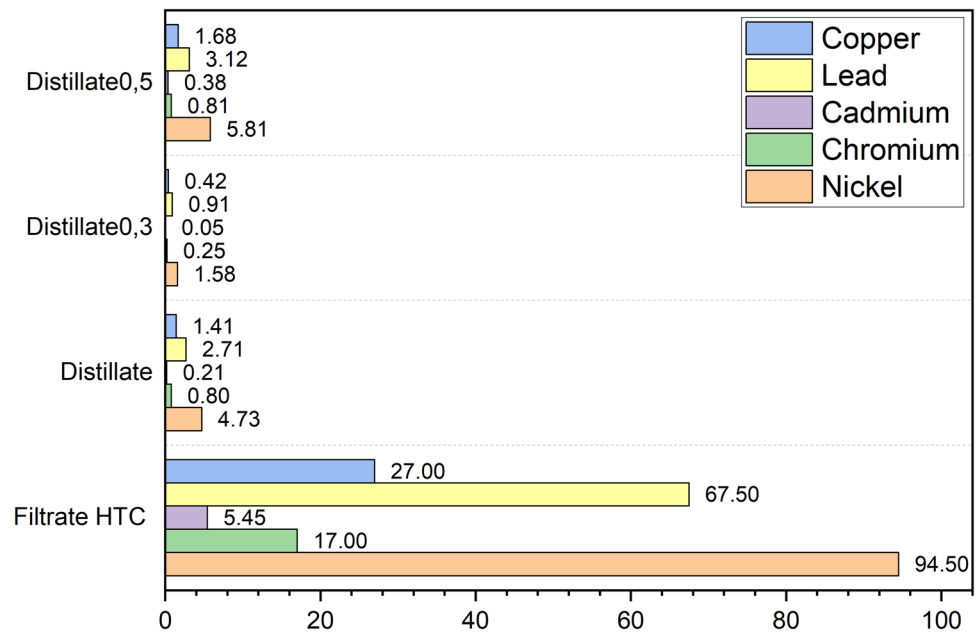


Fig. 8 Heavy metal contents in filtrate and distillates [mg/L]



The pH value increased after distillation, which indicates that the liquid has a more alkaline reaction using this method. The higher pH of the distillate suggests that most of the organic acids contained in the filtration remained in the distillation residue.

The presence of potential toxic compounds in HTC process waters has been reported in the literature. One of such compounds is phenol. The literature reports that the liquid after the HTC process has high concentrations of this compound, 633–666 mg/L [38]. In this case, the phenol content was not as high; the value was measured as 127 mg/L. However, distillation resulted in even lower levels of phenol

content. For distillation under atmospheric pressure, this value was 9.6 mg/L, which corresponds to a reduction of about 92%. At a pressure of -0.3 bar, a value of 8.0 mg/L was achieved (reduction of 94%). At a lower pressure of -0.5 bar, a value of 12 mg/L was reached, which corresponds to a reduction of around 91%.

The HTC process influences the migration of heavy metals from the solid phase to the liquid (Fig. 8). Most of the heavy metals remain in the hydrochar, but some may be found in the processing waters [39, 40]. This could pose a significant environmental problem. Consequently, very high contents of heavy metals, i.e. Cu, Ni, Cr, Cd, Pb, were

determined. These values were reduced by distillation of the filtrate after hydrothermal carbonization. For distillation at atmospheric pressure, the reduction of all heavy metals was 95–96%. When the pressure was lowered during distillation to – 0.3 bar, the reduction was higher at 98–99% for all tested heavy metals. When the pressure was lowered to -0.5 bar, the reduction was 93–95%. Pure heavy metals are characterized by high evaporation temperatures [41]. The high reduction of parameters was probably due to the fact that heavy metals could be present in the solution in their pure form and, thus, were not able to evaporate at this process temperature. The low content of these elements in the distillate and the hesitation of their content in distillates could be caused by local overheating of the liquid or the transfer of heavy metals deposited on water vapour particles.

Conclusions

The filtrated liquid phase from the hydrothermal carbonization of sewage sludge contains high-concentration contaminants and, therefore, requires an adequate method to facilitate its further disposal. The use of distillation processes to remove contaminants may be an appropriate method. Atmospheric pressure distillation enabled the removal of 95% COD and TOC, 99.5% of PO₄-P, 93% of Phenol, over 90% of heavy metals, and over 97% of free and total chlorine. It also generated an increase in pH and a decrease in conductivity. Higher reductions were obtained for distillation at a reduced pressure down to – 0.3 bar. In this case, distillation enabled the removal of 97% of COD, 98% of TOC, 99.9% of PO₄-P, 94% of Phenol, more than a 98% reduction in the value of heavy metals, and more than 98% of free and total chlorine. Lowering the pressure to – 0.5 bar did not cause any significant effects in the treatment of the liquid after the HTC process resulting in similar data to atmospheric pressure. Apart from the fact that the pressure reduction has produced a slightly higher reduction, it is important that the boiling point of the liquid is lowered at a reduced pressure. This will also have a positive impact on the economic aspects as lower process temperatures lead to energy savings and material savings in design. Further research is required regarding the performance of distillation purification of liquid from the HTC process.

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Data Availability The data that support the findings of this study are available from the corresponding author on reasonable request. The data

that support the findings of this study are openly available in RODBUK at <https://doi.org/10.58032/AGH/0RZXQK>.

Declarations

Conflict of Interest The authors declare no competing interests.

Ethical Approval Not applicable.

Consent to Participate Not applicable.

Consent for Publication Not applicable.

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Authors and Affiliations

Klaudia Czerwińska¹  · Maciej Śliz¹  · Małgorzata Wilk¹ 

✉ Klaudia Czerwińska
kczerwin@agh.edu.pl

Protection, AGH University of Science and Technology, 30
Mickiewicza Avenue, 30-059 Krakow, Poland

¹ Faculty of Metals Engineering and Industrial Computer
Science, Department of Heat Engineering and Environment