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The Role of Negative Emissions Technologies in 2050 Decarbonation Pathways
Jean-Pierre Deflandre and Florence Delprat-Jannaud (Guest Editors)

REGULAR ARTICLE OPEN ACCESS

Low-temperature and Low-pressure HydroThermal Liquefaction (L-HTL) of biomass using ultrasonic cavitation to achieve a local supercritical state in water

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Abstract. HydroThermal Liquefaction (HTL) is a process that involves the reaction of polymer compounds such as cellulose, lignin, synthetic plastics, etc. with near-critical or supercritical water to form low molecular weight liquid compounds, similar to natural oil which is believed to have formed over millions of years. Compared to other biomass recovery methods such as pyrolysis or anaerobic digestion, HTL is highly efficient with an energy efficiency of up to 90%, while the others have an efficiency of only around 30%. However, traditional HTL requires extremely high temperatures (250–450 °C) and pressures (100–350 bar), which are challenging to achieve using large-scale industrial equipment. This study proposes the use of ultrasonic cavitation to induce a supercritical state in water locally, rather than throughout the entire reactor, making it possible to perform HTL reactions using inexpensive and simple devices. The study demonstrates the successful conversion of pure cellulose to low molecular weight liquid compounds using potassium hydroxide as a catalyst.

Keywords: HydroThermal Liquefaction, Synthetic fuels, Biowastes.

1 Introduction

Despite differences in attitudes towards climate change, strategies for promoting a green revolution, the speed of transformation, and economic systems, it is now clear that abandoning fossil fuels as the primary energy source and transitioning to renewable energy sources within the next generation is essential for human survival [1]. Thus, finding and developing new renewable and sustainable sources of energy and raw materials is crucial [2].

While there are realistic solutions for securing electricity supply, such as wind, solar, and tidal energy, and new-generation nuclear reactors, the transition of the transport sector to renewable energy is not fully resolved. For most applications, fuel is required in liquid forms, especially in the transportation sector. Moreover, the energy density of liquid fuels is much higher than that of biomass [3].

Battery-powered cars, are expected to increase in use. Unfortunately, this alternative does not have a low CO_2 footprint — depending on the energy used to charge the batteries and especially the materials needed to make the car, an electric car has an environmental footprint of from 50% to more than 100% that of a petrol-fuelled car [4]. Moreover, it is not feasible to make battery-powered trucks or agricultural machinery due to the battery's required capacity and the materials needed for production [5, 6].

Hydrogen cars are being discussed as an alternative, but similar problems arise, such as expensive, heavy, and dangerous hydrogen tanks and low efficiency: on average [7], approximately one-third of the stored energy is returned. Air transport based on an alternative energy source is even more problematic. The only conceivable solution appears to be a molten salt reactor, but its development is likely decades away [8, 9].

Thus, in the short term, the most sensible solution is to produce green fuel for existing means of transport. Three different solutions exist: producing fuel from agricultural products (biodiesel, bioethanol, etc.), which competes with food production and destroys large-scale natural landscapes [10–12]; producing fully synthetic fuels, which requires a large amount of electricity in high demand worldwide [13], and converting unnecessary organic material into fuel. Biomass is the most substantial energy source, including forestry and agricultural residues, energy crops, peat production residues, and aquacultured algae, as well as all organic wastes such as sewage sludge, food, and other organic waste. More than 400 million tons of plastic waste is produced annually [14, 15]. If agricultural, forestry waste,

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sewage sludge, etc., are added, then the amount of discarded organic matter is several billion tons.

Two methods have long been used in biowaste recovery: pyrolysis and biological fermentation [16, 17]. In the first step of pyrolysis, the organic material is dried and heated to a high temperature (400–1000 °C) under anaerobic conditions. Some of the material becomes oil and combustible gas, and some remain in the reactor as biochar. Pyrolysis has limited use due to raw biomass's moisture content (which should usually be lower than 10%) [18] and relatively high oxygen content, making their utilization in internal combustion engines challenging; oxygen-rich oil products tend to polymerize rapidly when heated.

Fermentation uses anaerobic bacteria to convert some organic matter into methane, but both processes have low efficiency, with only up to one-third of the feedstock being converted into fuel in both cases [19, 20].

HydroThermal Liquefaction (HTL) is a viable alternative to these processes, allowing 85%–90% of organic matter to be converted into fuel. HTL is a chemical process similar to the one that has taken place at the depths of the Earth for millions of years, resulting in the formation of oil and natural gas [21]. The reaction takes place in the presence of supercritical water, as a result of which the long polymer chains decompose, hydrogenate, and depending on the catalyst, form low molecular weight oils and combustible gases (methane and others) [3, 22].

Technically, HydroThermal Liquefaction (HTL) is a thermal depolymerization process used to convert a mixture of biomass and other macromolecular organic material with water into crude-like oil (biocrude) in the presence of water in a liquid state or under supercritical conditions, effectively in a temperature range of 250–450 °C and pressures of approximately 100–350 bar [23–25]. During HTL processing, organic material undergoes a number of different reactions. They include dehydration, hydrolysis, and decarboxylation (see Fig. 1), as well as various repolymerization and condensation reactions.

These reactions yield a significant amount of liquid products that can be utilized as biofuel or serve as raw materials for producing plastics, among other things. HTL is a highly promising technology for the valorization and recycling of various forms of biomass, including biowaste. However, the extreme conditions, such as pressures and temperatures, required for this process remain a significant barrier to its widespread use. Although several setups for HTL equipment have been proposed, none have adequately addressed this challenge [3]. The current study seeks to resolve this issue by employing ultrasonic cavitation to create the necessary conditions locally, rather than throughout the entire reaction zone.

The primary products of HTL include biocrude, a crude oil-like liquid, and biochar. Gases containing CO_2, H_2, CH_4, and CO, as well as an aqueous phase containing dissolved salts and water-miscible organic compounds, including alcohols, ketones, phenols, acids, and some heteroatomic compounds, are also produced. The reaction patterns for different biomass types vary, depending on the process conditions, such as pressure, temperature, heating rate, and pH. The type of catalyst used is also a crucial factor.

Although biocrude generally has better quality than pyrolysis oil, the removal of oxygen and other heteroatoms, as well as the saturation and cracking of hydrocarbons, must be achieved to meet statutory requirements for motor fuel quality. This is typically accomplished through catalytic hydrogenation (hydrotreating) [3, 22].

Therefore, HTL offers a promising solution for the valorization and recycling of various forms of biomass, including biowastes. However, the need for extreme conditions remains a significant obstacle, which has not been adequately addressed by any of the proposed HTL equipment setups. The current study overcomes this problem by utilizing ultrasonic cavitation to create the necessary conditions locally, rather than throughout the entire reaction zone.

Cavitation is a physical process in which rapid changes in pressure cause small cavities filled with vapor to form in areas where the pressure is relatively low [26]. These cavities, or bubbles, collapse and generate a strong shock wave upon entering areas of higher pressure. This process creates very high energy densities, local temperatures, and pressures (up to 5000 K and 2000 atm) [27, 28] within limited volumes for a brief period. As a result, the overall material remains at the previous conditions. Cavitation waves allow for supercritical conditions to be achieved in parts of the liquid, while the bulk volume of the liquid remains at moderate parameters. In addition to the thermal and pressure effects of the imploding cavitation bubbles, other mechanisms have their effects. Shockwaves and microjets are also created by cavitation bubbles, affecting the solid and gel phases mechanically, and causing shear forces. The movement of bubbles creates turbulence and agitation, which, in turn, intensifies the heat and mass transfer within the reacting mixture. Other physical effects of ultrasound on water-based pulps include direct and indirect heating, acoustic streaming, and nebulization. An implosion of a cavitation bubble can be seen as an adiabatic compression process, which causes an abrupt local increase in temperature at a rate of 1000 °C/s. Although the acoustic cavitation weakens with increasing temperature, other effects persist. A very important factor is radical reactions; H•, OH•, and HO_2• radicals are generated from water and a wide variety of organic radicals is generated from organic matter, which readily attack the organic molecules and aid in the cleavage process of large polymeric chains into smaller size molecule [29–31].

Only a low additional pressure (up to 15 bar) is required to prevent water and reaction products from boiling at working temperatures (up to 250 °C). The energy required for HTL reactions is provided by cavitation waves, which increase the temperature and pressure within small volumes of liquid, thereby inducing supercritical conditions within these volumes of water.

The role of the catalyst in HTL varies depending on the process temperature, retention time, and reactor configuration. At moderate temperatures (up to 350 °C), catalysts accelerate the formation of biocrude and increase the yield. Heating beyond the supercritical point contributes to the improved removal of heteroatomic compounds from the biocrude [18].

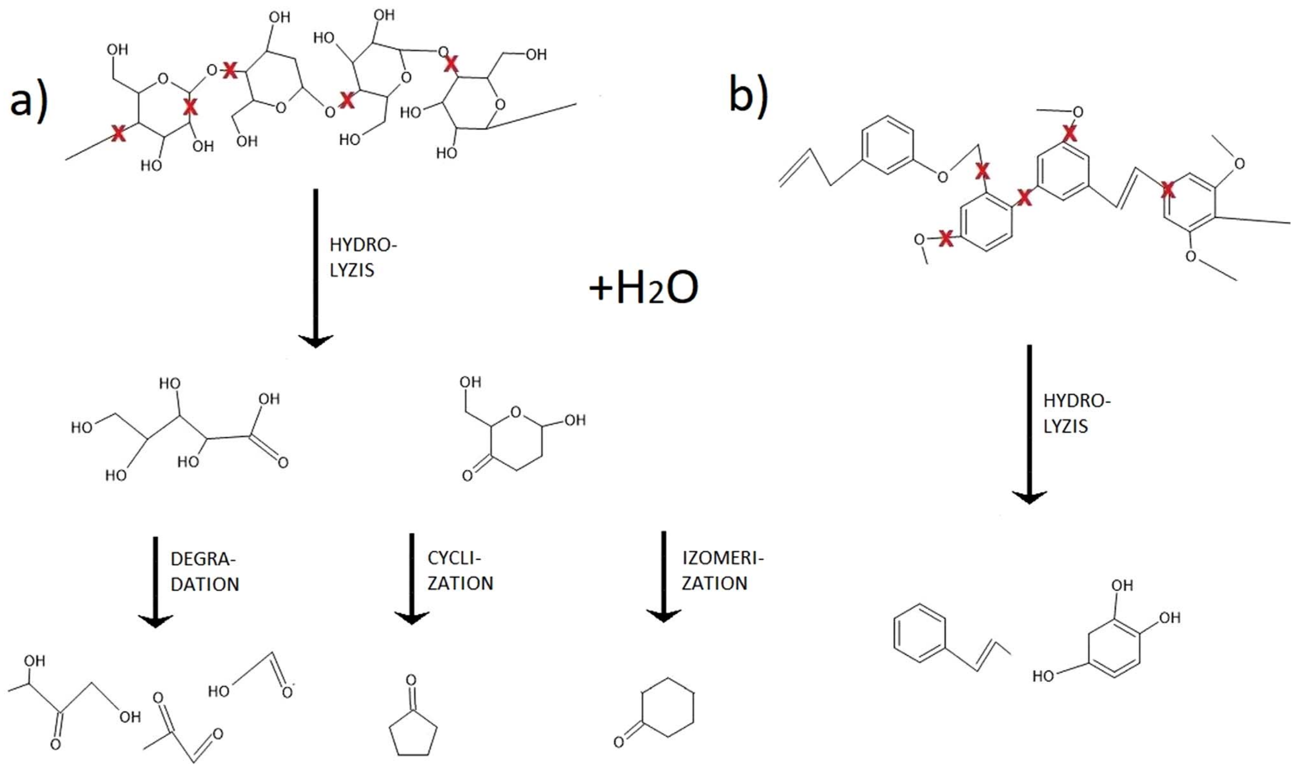

Figure 1. Some possible pathways for degradation of cellulose (a) and lignin (b) in the HTL reaction.

Catalysts can be classified into two main types: homogeneous and heterogeneous [9]. Homogeneous catalysts include alkaline salts, hydroxides (such as NaOH and NH_4OH) [24], and mainly organic acids, while heterogeneous catalysts include transition metals like Ni, Cu, Fe, metal oxides, activated carbon, zeolites, clay minerals (such as montmorillonite [32]), and composite catalysts (such as $CoMo/Al_2O_3$).

Ciuffi *et al.* [24] demonstrated that different ionic homogeneous catalysts (Na^+ and NH^{4+}) induce different reaction mechanisms for lignin degradation. Ammonia facilitated the formation of catechols, while the use of Na^+ resulted in significantly higher phenol content in HTL products. The authors proposed an ionic pathway for lignin depolymerization.

The thermal energy recovery of the HTL process can be measured in terms of the energy ratio, which is defined as follows [18]:

$$\mathrm{ER} = \frac{\mathrm{Y}_{\mathrm{biocrude}\,\times}\mathrm{CV}_{\mathrm{biocrude}}}{\mathrm{CV}_{\mathrm{feedstock}}}.$$

Here, ER represents the energy recovery ratio, $\mathrm{Y}_{\mathrm{biocrude}}$ is the biocrude yield, $\mathrm{CV}_{\mathrm{biocrude}}$ is the calorific value of biocrude, and $\mathrm{CV}_{\mathrm{feedstock}}$ is the calorific value of the feedstock.

Regarding energy recovery, all homogeneous catalysts are efficient compared to no catalyst, except for a few cases involving mainly cellulosic materials as feedstock. Alkali catalysts are generally more efficient than acidic catalysts. Carbonate salts of alkali metals and ammonium usually result in higher energy recovery than their respective hydroxides [18].

Figure 2 depicts a hypothetical pathway for the degradation of cellulose.

2 Methodology

Currently, the workgroup is in the process of constructing a fully functional lab-scale HTL flow reactor with all the necessary features for pumping the reaction mixture and controlling the reaction parameters such as temperature, pressure, flow rate, separation of reaction products, and distillation. However, due to budget constraints, a high-capacity ultrasonic cavitation device (20 kW) could not be acquired. To conduct proof-of-concept experiments, we used an ultrasonic homogenizer from Hielscher, which is designed for laboratory studies of pharmaceuticals. It was equipped with logging capabilities for power consumption, temperature, and pressure. A block sonotrode BS4d18 (Hielscher Ultrasonics) with a booster B4-1.8 and sonotrode adapter A2Clamp_FKMm, the sonotrode was fed by the industrial ultrasonic processor UIP1000hdT-230 (1000 W, 20 kHz). The experimental apparatus is shown in Figure 3.

In its original configuration, the ultrasonic homogenizer did not lead to any significant degradation of cellulose, as the ultrasound dissipated in a large volume. To address this issue, we designed an acoustic concentrator that could focus the ultrasound into a narrow 1–2 mm gap. We also developed a peristaltic pump with a working element made of heat-proof silicone.

Pure cellulose ASPEN 350/8UB from Estonian Cell (Heinzel Group) was used as a raw material to avoid the

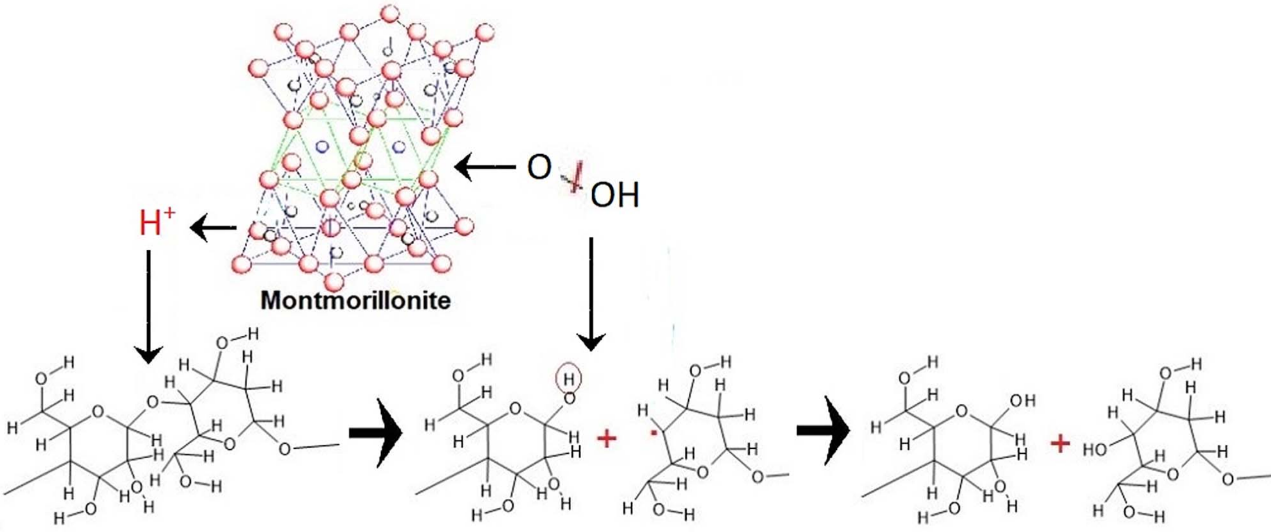

Figure 2. Possible pathway for catalytic degradation of lignin.

Figure 3. Setup of the reaction chamber.

presence of contaminants in the reaction mixture. The dry cellulose was ground and mixed with water (3 wt% of cellulose) before adding 3% KOH as a catalyst.

If the mixture that has flowed through the device is left to stand, a lighter layer separates on the surface (Fig. 4). It consists of a mixture of very different low molecular-weight substances.

If left standing longer, it splits into several layers (Fig. 5).

Spectrum BX II Fourier transforms infrared spectrometer (Perkin Elmer) with ATR (ZnSe) device was used in the FTIR analyses.

Dichloromethane extracts of the reaction mixture were dried over magnesium sulfate and FTIR spectra were measured directly after evaporation of solvent on ATR crystal. The evaporation was determined by the disappearance of the C–Cl stretching vibration signal at 740 1/cm.

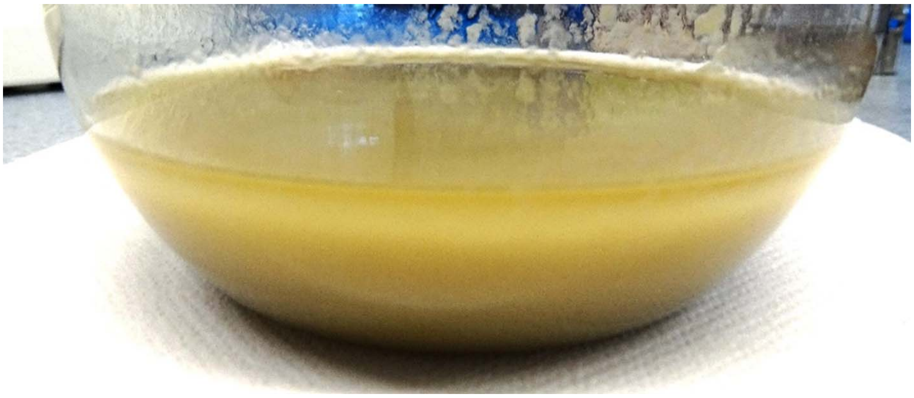

Figure 4. Result of the reaction.

Figure 5. Mixture split into different layers.

3 Results and discussion

Firstly, it should be noted that the sound power we used (1 kW) apparently proved insufficient for carrying out the HTL reaction in our experiment. Using the original flow chamber purchased, we observed no changes in the reaction mixture.

Subsequently, we designed an acoustic concentrator to produce a planar wave, after which low molecular weight components emerged in the reaction mixture. This can be taken as evidence of the occurrence of the HTL reaction in the mixture, as all parameters remained unchanged, including the acoustic energy transferred to the mixture. By concentrating the acoustic energy into a smaller space between acoustically mirroring surfaces, we immediately observed the emergence of components with lower molecular mass.

However, the acoustic power we utilized was not sufficient for carrying out the HTL reaction throughout the mass of cellulose particles, and only the surface layer of the particles reacted with a single sonication. In order to achieve the HTL reaction throughout the whole volume of the mixture, we circulated the mixture using a peristaltic pump until cellulose particle gelification was observed. Although the energetic efficiency of our current technical setup was low due to most of the ultrasonic energy being consumed to heat the liquid phase, we calculated the efficiency using the logging data of the ultrasound controller.

It is necessary to have catalysts present for the HTL reaction to occur. We tested a variety of different catalysts, and these experiments are currently ongoing, as our low-power laboratory apparatus allows for easy comparison of different catalysts.

Spectrum BX II Fourier transform infrared spectrometer (Perkin Elmer) equipped with an ATR (ZnSe) device was used for conducting FTIR analysis. To obtain the spectra, we dried dichloromethane extracts of the reaction mixture over magnesium sulfate and measured them directly on the ATR crystal following the evaporation of the solvent. We confirmed the completion of evaporation by observing the disappearance of the C–Cl stretching vibration signal at 740 cm^{-1}.

The FTIR spectra of the process extract revealed the presence of a mixture of new compounds that were formed during the treatment of the cellulosic material.

Figure 6 shows the FTIR spectrum of cellulose treated in the reactor, the spectrum of untreated cellulose is shown for comparison. We can see that a lot of new reaction products have formed in the mixture. First of all, the signals at 2932 cm^{-1} (several signals), 1464 cm^{-1}, and 1376 cm^{-1} indicate the formation of aliphatic compounds, *i.e.* the degradation of cellulose into low molecular weight chains.

The formation of aliphatic compounds is easily explained by the hydrolysis of cellulose, the possible course of which is shown in Figure 7.

The intensive signal at 1044 cm^{-1} may also correspond to C–O stretching vibrations of the primary aliphatic OH group [33] (see Fig. 8).

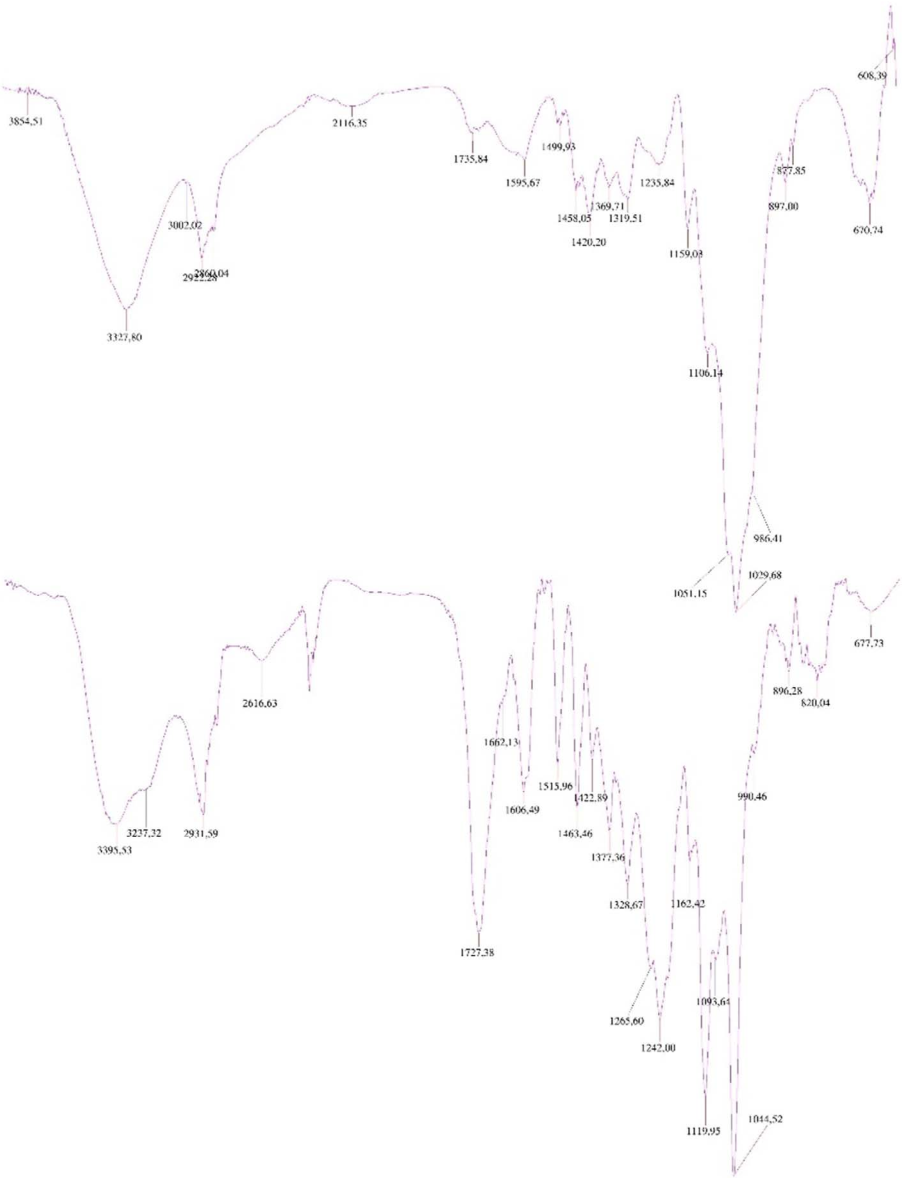

Figure 6. FTIR spectra of untreated cellulose (up) and water extract after cavitation (down).

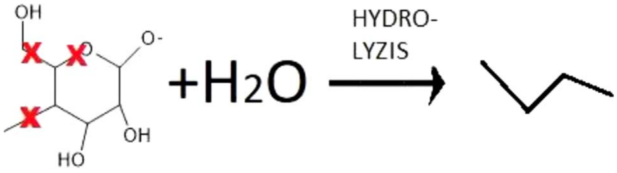

Figure 7. Possible formation process of aliphatic compounds.

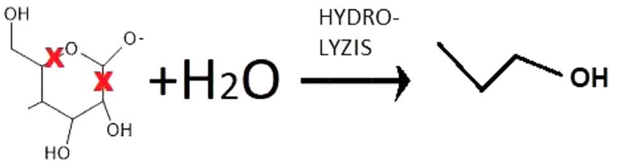

Figure 8. Possible formation process of aliphatic compounds with primary OH group.

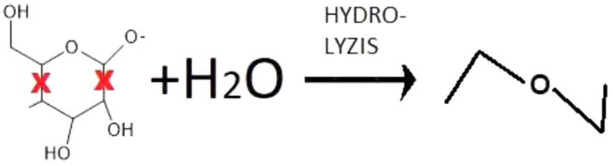

Figure 9. Possible formation process of C–O–C bond.

A signal at 1727 cm^{-1} is due to stretching vibrations of the C=O bond and together with the vibrations of C–O–C at around 1242 cm^{-1}, and 1120 and/or 1045 cm^{-1} correspond to ester groups (see Fig. 9). They can form similar to the previously described processes when the oxygen atom remains in the middle of the chain.

The signals at 1606, 1516, and 1425 cm^{-1} and several signals around 820 cm^{-1} belong to an aromatic ring and

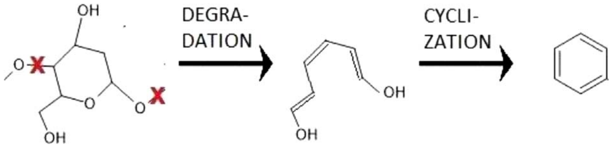

Figure 10. Possible formation process of aromatic compounds.

C_{Ar}–Hoop vibrations, respectively. The broad signals of OH and C–O vibrations at 3200–3400 cm^{-1} and 1230–1270 cm^{-1}, respectively, confirm the presence of phenolic compounds. This is also supported by the existence of the aromatic structures.

The process of formation of aromatic compounds is more complicated. As a possible explanation, we see that the carbon chain loses some hydrogen atoms and OH groups, resulting in the formation of double bonds. As a result of the cyclization of such a chain, an aromatic carbon ring is formed. A hypothetical reaction diagram is shown in Figure 10.

4 Conclusion

Our reaction setup and procedure enable the performance of an HTL reaction under moderate conditions, eliminating the need for very high pressures. We demonstrated the partial degradation of cellulose through the application of ultrasonic cavitation to achieve a supercritical state of water.

While significant work remains before this technology can be industrially applied, we believe that economically competitive HTL technology can revolutionize the world's fuel economy by facilitating a smooth transition from fossil fuels to biofuels. This technology has the potential to produce fuel suitable for existing vehicles and engines from 100% renewable sources, reducing greenhouse gas emissions from fossil fuels. The biocrude produced by HTL plants can be further processed in existing oil refineries, thus minimizing the costs of technological transition and preserving economic growth during the revolution.

Currently, vast quantities of biological waste are land-deposited or incinerated without any useful application, resulting in unnecessary air pollution and greenhouse gas emissions. Our advanced HTL technology has the potential to convert various types of biowastes, plastics, and tires into value-added products, including motor fuels and other items traditionally derived from crude oil. Alternatively, biowaste is treated to produce low-value products such as compost or inefficiently used as cheap fuel with low calorific value.

This technology can significantly contribute to the economic growth and social advancement of developing countries dependent on imported fuels, while also reducing the West's dependence on fossil fuel imports from unstable countries with authoritarian regimes, ensuring energy security and decreasing foreign trade deficits.

HTL technology produces minimal waste, and biochar also has several potential applications. CO_2 captured from any source can be used as a substrate for aquacultured algae, which, in turn, is an ideal raw material for HTL. Therefore, HTL technology has the potential to advance the transition to a circular economy by positively impacting numerous aspects of production and energy use.

Conflicts of interest

The authors declare that there is no conflict of interest.

Data availability statement

Data are available on request from the authors. The data that support the findings of this study are available from the corresponding author (J. Liiv), upon reasonable request.

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Author contributions

Conceptualization and methodology were done by J. Liiv; equipment design by N. Vaino, experimental and investigation by E. Rikmann; chemical analyses and data curation by U. Mäeorg, writing — original draft preparation by J. Liiv and E. Rikmann; writing — review and editing by U. Mäeorg and N. Vaino, supervision and project administration, by J. Liiv. All authors have read and agreed to the published version of the manuscript.

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