A TOILET SYSTEM BASED ON HYDROTHERMAL CARBONIZATION

E. Danso-Boateng, R. Holdich, A. Wheatley, <u>S. Martin</u>, M. Sohail, and D. Gyi

Loughborough University, Leicestershire, LE11 3TU United Kingdom

ABSTRACT

We are developing a toilet system that converts faecal material to an aqueous suspension of carbonised material that is safe to handle, and readily separated from the remaining liquid. It will also extract useful salts from the liquid. The system is aim to be the new generation universally appealing toilet and it will be of particular and urgent interest to areas where no (or very crude) sanitation exists. The system is designed to be self-sufficient in terms of energy input and to scale for a number of users in the range a few tens to a thousand or more. In parallel with our engineering development we are designing the system to provide users with a positive and comfortable experience.

Hydrothermal carbonisation (HTC) has received attention recently as a way to convert biomass - including sewerage - into coal like material. It involves heating the start material in water at high temperatures and pressures. Depending on the conditions used the process can produce hydrocarbon gases or liquids or coal like particles. Most HTC work involving sewerage treatment is currently aimed at replacing established large-scale treatment plants i.e. for places with well-developed sewerage services. Our system, using HTC, is aimed at bringing the process at a decentralized household (including combination of households) level so that the new generation toilet become accessible to all and particularly to those areas where none currently exists. Using HTC for toilets on continuous basis for such a small scale is the key innovation of our proposed system.

We will describe our work to characterise the energetics of the HTC process in order to optimise the process in terms of total energy input and how we have used this information to develop a continuous system based on a plug flow reactor. The material produced by the system can be easily separated from the remaining liquid and used to generate heat and power (via a generator including solar) in order to maintain the process. The solids are safe to handle, and look, feel and smell much like coffee grounds. In situations where electrical (or renewable energy like solar) power is available the solid material can be used either as a fuel for heating and cooking, as a soil conditioner and possibly for carbon capture. We will discuss the possibility of including other waste material (food, sanitary waste etc.) into the system.

In order to minimize the amount of water required to flush material away from the toilet bowl and to help maintain high sanitary standards we have been investigating anti-fouling coatings for the system. We will describe the results of studies of a nano-coating based on a responsive polymer that significantly enhances the rate at which water flushes away material that adheres to the toilet surface.

Keywords: HYDROTHERMAL-CARBONISATION, SEWAGE

INTRODUCTION

In this paper we describe our work developing a toilet system for the Reinvent the Toilet Challenge from the Bill and Mellinda Gates Foundation. This challenge aims to develop toilet systems that are able to deal effectively and cost-efficiently (less than $0.05 per person per day) with human waste in order to bring safe and affordable sanitation to those who currently do not have it Gates (2011). The systems must be self-sufficient in terms of water input and output and essentially self-sufficient in terms of electricity usage. The challenge further states that the process used in the system should process the material in around one day— this essentially precludes the use of techniques such as anaerobic digestion. In order to meet this challenge Loughborough University put together a team covering a wide range of academic expertise.

The concept we describe here is based on a process called Hydrothermal Carbonisation (HTC) (Funke et al. (2010); Libra et al. (2011)). In this process water at high temperature and pressure converts biological material into a coal like material. The products of the process can be used as fuel to maintain the process allowing our system to be self-sufficient in terms of energy input. The liquid produced by the system may be treated using ion exchange to remove salts. In order to minimise the amount of water used for processes such as flushing we intend to use a novel anti-fouling coating that is based on hydrophilicity rather than the more usual hydrophobic processes. In order to ensure that the system provides a pleasant experience for as many users as possible inclusive design based on ergonomics principles will be used.

Hydrothermal carbonisation is a process that converts biological material into coal like material through the action of water under moderately high temperatures and pressures. The process was originally developed in the early twentieth century as a model of the production of coal and oil like materials on geological timescales (Berl and Schmidt 1932). It has been the subject of sporadic research interest in the intervening years. More recently it has received attention as a means of CO_2 sequestration and as method of producing a soil-improving agent similar to the terra preta materials used by indigenous populations in regions such as the Amazon (Titirici et al. (2007); Costa et al. (2004). A key advantage of the HTC process for the RTTC is that it is exothermic and provided that the input material is suitable (not too diluted by urine, flush water, and other liquids-one possibility is to combine organic waste to improve suitability) then the heat generated is sufficient to make the process self-sustaining. We anticipate that more energy will be generated than is needed by the process

In the HTC process, the biomass is in an aqueous suspension throughout the reaction under increased pressure in a high-pressure reactor producing solids (char) or, liquid (water and water soluble organic compounds) and gases (mainly CO_2). The HTC process occurs at temperatures of 180◦C to 250◦C with the biomass feedstock totally submerged in water under autogenous pressures. The high pressures used ensure that the water remains in a liquid state throughout the process. The temperature and pressure used has an effect on the state of the products of the reaction (Libra et al. (2011)). For temperatures up to 200◦C (pressures up to 2 MPa) little gas is generated and most of the products are solids. At temperatures up to 400 ◦C more liquid hydrocarbon liquids and gases are produced. If the temperature/pressure conditions are in the supercritical range for water then the products are essentially all gaseous.

HTC is beginning to be used for the treatment of sewage on the scale of plants capable of treating the sewage output of small towns. These plants use grid electricity to power the system. We have been investigating whether the method can be scaled down to smaller populations—ideally family sized groups, but up to a hundred or so is still within the RTTC criteria—and whether at these scales enough fuel and energy is released by the HTC to make the process self-sufficient. For ease of handling, a lot of our initial work in this area has been based on a simulated faeces/water mixture. However we have also investigated the conversion of primary waste sludge from a local sewage plant.

An important consideration in the energy balance of the HTC process for our system is the heat capacity of water (4.2 J g−1 K−1 (Blokhin et al. 2011)—about four times the value of materials such as cellulose. This means that a lot of energy will be tied up in heating the water component of the mixture that undergoes HTC. In order to minimise the amount of water input to the system for processes such as flushing we have investigated the use of a block-co-polymer system that creates an anti-fouling layer on the surface of a wide range of materials. Any material on the surface of the coating is washed away easily as the polymer coating forms a hydrophilic layer on the coated surface and flush water is drawn under the contaminant and is lubricated away.

EXPERIMENTAL

Faecal simulant was formulated following the recipe by Wignarajah et al. (2006). This comprised 37.5% cellulose, 37.5% yeast, 20% peanut oil , 4% KCl, 1% Ca(H2PO4) (all purchased for Sigma-Aldrich, UK). Co-mix anaerobic digested (AD) sewage sludge was acquired from Wanlip Sewage Works in Leicester in a sealed

container. The sludge contained 4% solids (96 % moisture). The sludge was kept in the cold room of the Civil Engineering Water Laboratory throughout the experiment.

Batch HTC of the faecal simulant was conducted in a 250mL stainless steel reactor. Carbonisation was conducted by loading weighed materials separately into the reactor, mixed and thereafter 50 % by proportion of the water added and mixed to from a paste. The remaining water was added to suspend the solids, and then the reactor was sealed. Solid loadings of 25, 15 and 5 % (wt) were used in separate tests. The initial pressure of the vessel was set to 0.3 MPa before the reactor was immersed in the oil bath (B7 PHOENIX II, Thermo Scientific, UK) and heated to 200 ∘C for 30, 50 minutes, 1, 1.5, 2 and 4 hours. The solid (hydrochar) was separated from the liquid (dewatered) via a vacuum filter (20 μm to 50 μm Whatman). The initial time taken for the reactor content to reach the 200 ∘C reaction temperature was noted. The average heat-up time ranged from 10 min to 20 min. Final pressures were between 1.4 MPa to 1.71 MPa. In order to determine the effect of the heat-up time on the solid yields of each test (i.e. each temperature run), the reaction was stopped immediately at heat-up time and the contents dewatered to obtain the solids as above.

The solids were then dried in an oven at 55 ∘C for 24 h to remove any residual moisture and thus determine the solids left after carbonisation. The kinetics of the HTC process were followed by assuming that the mass of converted follows first-order kinetics according to:

$$m_{char} = m_0 \exp{-kt} \qquad [1]$$

where m_{char} is the mass of hydrochar produced, m_0 is the initial mass of solids, k is the kinetic rate constant and t is the time that the process ran for. A standard Arhennius analysis was used to extract the activation energies for the simulant and AD materials from the rate constant data. The carbon, hydrogen, and nitrogen contents of the feedstock and the carbonised solids were measured with a CE-440 Elemental Analyzer (Exeter Analytical Inc., UK).

Two candidate antifouling coating materials were supplied by Chamelic Ltd. (UK) these are referred to as Cham-1 and Cham-3 in this paper. The details of their structures are commercially confidential however they are block co-polymers in which one block is hydrophilic and the other is hydrophobic. In the samples provided the hydrophilic blocks were polyelectrolytes of opposite charges. Both polymers were processed by dissolving them in water-methanol mixtures. Clean glass slides were coated in the materials by dropping on the solutions and allowing them to dry in air at room temperature.

The contact angle of water drops in contact with the coated slides were measured using a Dataphysics OCA instrument. A sessile drop of water was held in contact with the surface being tested and the contact angle at the left and right hand sides of the drop were measured and averaged. These measurements were made immediately after coating the glass and over a period of seven days to investigate the stability of the coating.

The anti-fouling properties of the coatings were assessed by applying peanut butter to coated slides and washing them under flow rates of water typical of western style flush toilets.

RESULTS AND DISCUSSION

The rate constant analysis showed that the activation energies for the char and AD material were xx.x1 and 72.8 kJ mol^{-1} respectively. These results show that the simulant is a reasonable substitute for the AD material. In operation the material entering the carbonisation system is likely to have undergone less aerobic digestion than the AD material used in our experiments.

The CHN measurements showed similar changes to those reported elsewhere (e.g. Berge et al. (2011)) the raw AD material contained 43% carbon, 6.5% hydrogen and 3.8% nitrogen. The relative amount of carbon increased with increasing process temperature. After processing at 200∘C the CHN content of the AD material were: 44.5%, 6.0% and 1.8% respectively.

Our initial plan was to develop a HTC system that worked as a batch process. Once sufficient material had been obtained then the system would be pressurised (possibly with a hand pump to minimise energy requirements or using renewable energy like solar) and the carbonisation process initiated. At the end of the process the pressure would be rapidly released causing most of the water to flash-evaporate and the solids would be collected to become the fuel to be burned to generate the heat needed for the next cycle. Whilst developing this concept it was realised that a continuous flow system could bring significant advantages to the process. In particular there will be no open and closing of a pressure vessel bringing an overall mechanical simplicity and an enhanced safety. A diagram of the system is shown in Figure 1. The main parts are a collection tank, a macerating pump, a multi-pass reaction vessel, and a flash evaporation system. The waste material (urine, faeces, and flush water) is pumped from the collection tank into the reactor vessel. The macerating pump provides the initial pressure to the system but the heating causes the pressure to rise to that required for the reaction to proceed. In our prototype system the reactor vessel is a three-pass design however this will be optimised in further work. The flow rate is set so that the material spends about 15 minutes inside the reactor vessel. After this the pressure is released in a flash vessel. A portion of the water immediately turns into steam taking with it volatile organic compounds. This steam is collected and used to pre-heat the material in the storage tank. This gives rise to a significant saving in heat energy. After passing through the collection tank the remaining gas is collected and as it contains volatile organic compounds it can be fed back into the fuel stream. The liquid/solid stream from the flash vessel is filtered using a microslot filter. The solids are collected either for use as a fuel source or as a soil enhancing agent. The remaining liquid will contain a variety of low molecular weight carbon based compounds. This liquid will be treated in an anaerobic digester in order to generate methane fuel. The small size of the molecules in the water means that the time scale for this process should be of the order of a couple of hours. Once this process is complete the remaining liquid will be safe to dispose of. Ideally the amount of volatile materials generated by the process will provide sufficient heat for the process. This will allow us to use a simple gas burner arrangement as the heat source. The amount of volatile material produced by the process can be enhanced by operating at higher temperatures (and hence pressures).
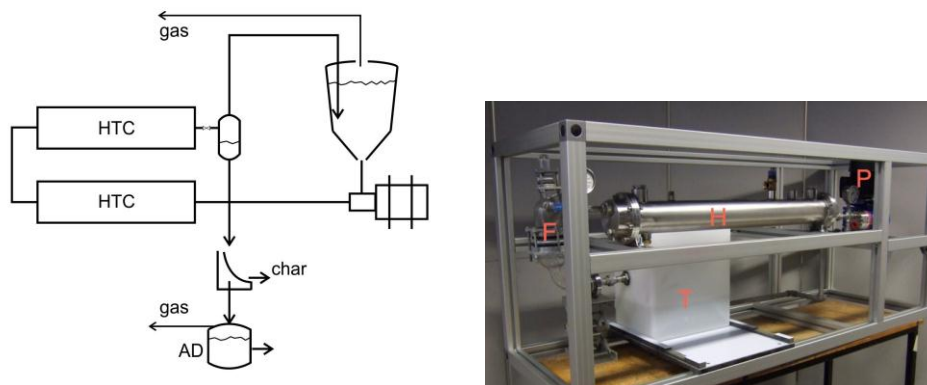

Figure 1. Schematic of continuous flow HTC system (left hand side), prototype conversion system (right hand side)—P-pump; H-HTC conversion vessel; F-flash chamber; T-tank.

In order to better understand the energy requirements for the process we have performed simple calculations of the energy deficit/surplus based on the energy required to heat water to process relevant temperatures and pressures and the energy available from combustion of the input solids. The amount of energy available from the faecal material is going to depend on the diet of the users of the system. Literature values indicate that a value of 17 MJ kg^{-1} is reasonable (Otero et al. (2002)). Using a spreadsheet we are able to calculate the energy balance for a full range of solid contents. We will report a more detailed description of these calculations in a further publication. The simulations show that net energy is produced by the process for solid contents as low as 5%. At low solid content levels the energy surplus is marginal and it is likely that all the solids will be required as fuel in addition to the volatiles and methane produced. We are investigating methods of increasing the solid content of the mixture going through the system. Another route to providing sufficient energy for the process is to include extra organic material such as kitchen waste in the HTC process.

For Cham-1 coated glass the wetting effect of the layer was so strong that the water was completely spreading over the length of the test and no contact angle could be measured. For Cham-3 coated glass the initial contact angle was 12.5°(±5°). After one week the coating had been applied this value had risen to 13.2°(±2.5°). For reference the contact angle of water on clean glass was measured to be 13.6°.

The results of typical wash tests are shown in Figure 2. The figure shows that the Cham-3 coated glass was a much more effective as an antifouling coating than either the Cham-1 coated glass or the uncoated glass. It must be borne in mind that faecal matter may interact differently with the coating. This means that further, more realistic tests are necessary before deciding which coating is better in the toilet cleaning application.
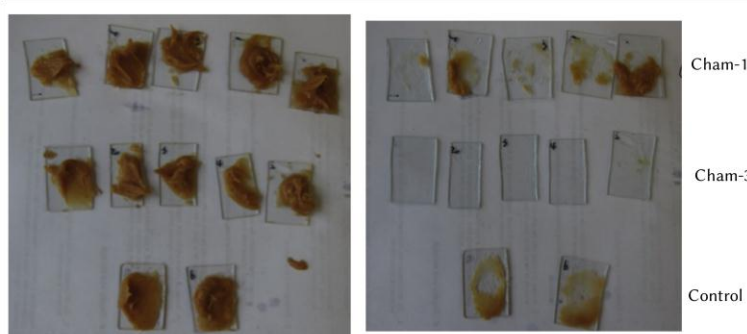

Figure 2. Antifouling effect of the Cham-1 and Cham-3 coatings. The left hand image shows the samples after coating with peanut butter. The right hand image shows the result of washing the samples in running water. See text for further details.

Now that we have established the viability of the HTC process for the RTTC system we are going work towards a fully operational prototype. There are two key streams to this development. We will optimise the processing system so that the energy losses are minimised and that as compact a system as possible is built. We will also make sure that the toilet part of the system is useable by as large a range of the target population as possible and that it provides a pleasant user experience as possible. The continuous flow system that we are proposing is most readily optimised for user numbers of a hundred or so (i.e. communities) in the short term. We intend to target this order of user numbers for our initial development. Once the technology is established we intend to move to developing the processing so that it can operate at the level of individual households.

ACKNOWLEDGEMENTS

REFERENCES

Berge, N. D., Ro, K. S., Mao, J., Flora, J. R. V., Chappell, M. a., and Bae, S. (2011). Hydrothermal carbonization of municipal waste streams. Environmental science & technology, 45(13):5696–703.

Berl, E. and Schmidt, A. (1932). Über die Entstehung der Kohlen. II. Die Inkohlung von Cellulose und Lignin in neutralem Medium. Liebigs Ann. Chem., 493(1):97–123.

Blokhin, A. V., Voitkevich, O. V., Kabo, G. J., Paulechka, Y. U., Shishonok, M. V., Kabo, A. G., and Simirsky, V. V. (2011). Thermodynamic Properties of Plant Biomass Components. Heat Capacity, Combustion Energy, and Gasification Equilibria of Cellulose. Journal of Chemical & Engineering Data, 56(9):3523–3531.

Costa, M. L. D., Kern, D. C., Pinto, A. H. E., and Souza, J. R. D. T. (2004). The ceramic artifacts in archaeological black earth (terra preta) from lower Amazon region, Brazil: mineralogy. Acta Amazonica, 34(2):165–178.

Funke, A., Ziegler, F., and Berlin, T. U. (2010). Hydrothermal carbonization of biomass : A summary and discussion of chemical mecha- nisms for process engineering. Society, pages 160–177.

Gates (2011). Water, Sanitation & Hygiene.

Libra, J. a., Ro, K. S., Kammann, C., Funke, A., Berge, N. D., Neubauer, Y., Titirici, M.-M., Fühner, C., Bens, O., Kern, J., and Emmerich, K.-H. (2011). Hydrothermal carbonization of biomass residuals: a comparative review of the chemistry, processes and applications of wet and dry pyrolysis. Biofuels, 2(1):71–106.

Otero, M., Dã, C., Calvo, L. F., Garcã, A. I., and Morã, A. (2002). Analysis of the co-combustion of sewage sludge and coal by TG-MS. 22:319–329.

Titirici, M.-M., Thomas, A., and Antonietti, M. (2007). Back in the black: hydrothermal carbonization of plant material as an efficient chemical process to treat the CO2 problem? New Journal of Chemistry, 31(6):787.

Wignarajah, K., Litwiller, E., Fisher, J., and Hogan, J. (2006). Simulated Human Feces for Testing Human Waste Processing Technologies in Space Systems. In International Conference On Environmental Systems. SAE.