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Studies on influence of process parameters in upgradation of bio-oil derived from HTL of domestic household waste: Application of response surface methodology

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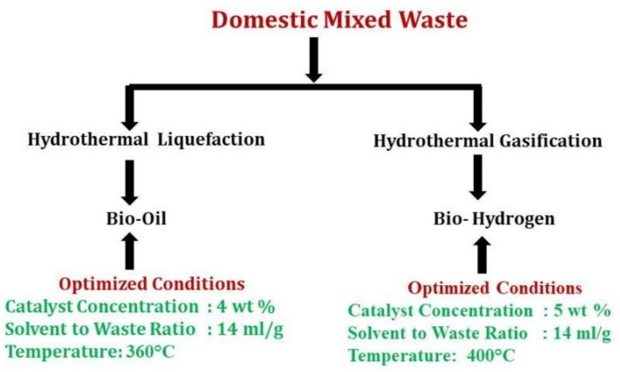
Graphical abstract



Abstract

This research focuses on hydrothermal gasification (HTG) and hydrothermal liquefaction (HTL) studies to produce bio-hydrogen from domestic mixed waste. HTG and HTL studies were studied at temperatures of 300–450°C and 300–400°C, correspondingly, with a catalyst level of 6 wt %. The sol-gel technique was used to make the Bentonite/Nb-TiO_2 catalyst. For a solvent-waste proportion of 14 millilitre/gram, an C_2H_5OH - water proportions of 2:2, and a period of one-hour, maximum H_2 output from HTL was 30 wt % (catalyst loads: 4 wt %) and HTG was 40 wt % (catalyst loads: 5 wt %). As C_2H_5OH acts as half-solvent and ideal solvent to bio-mass capacity it shot up H_2 result in the HTG procedure by methanation, gas water shift and improving responses in the organization. The second output of the HTL procedure was bio-oil, which yielded 35 wt % with O/C as well as H/C standards of 1.2 and 1.0, respectively, and an HHV of 42 Mega Joule/kilogram. In the HTL system, mechanistic procedures such depolymerization, hydration, reduction, and hydrolysis generated in a larger proportion of gaseous product. Thermal and the solvent-to-waste proportion is the ideal factors in the H_2 synthesis procedure, according to experimental analysis. Energy nexuses from domestic mixed trash are taken over in this research.

Keywords: Domestic waste, bio-oil, RSM, HTG, HTL

1. Introduction

Bioenergy has been viewed as a potential solution to exhaustible fossil fuels in terms of reducing greenhouse gas emissions. For dry biomass, pyrolysis and air gasification are used. As an outcome, there is an upsurge usage. Hydrothermal alteration methods are chosen for valorizing damp organic material to useful biofuel in part to avoid this costly procedure. When related to fossil fuels, H_2 energy is a pure, economical, and sustainable source. The Fischer-Tropsch method can be used to directly or indirectly generate compounds or fuels using H_2 energy. H_2 was produced on a large scale by steam transforming CH_4. Methanation, Gasification, Partial Oxidation, Involvement of photoautotrophic algae and Reforming are some of the other techniques for producing H_2 (Arun *et al.*, 2020). Subcritical and supercritical water gasification are two environmentally preferred ways employed in producing hydrogen from high moisture biomass. Thermochemical process, Hydrothermal gasification (HTG) converts biomass into gaseous products in freshwater systems (Sztancs *et al.*, 2020). HTG was the preferable approach for decomposition of biomass with greater moisture content because it was done beyond the critical point of H_2O. Biomass, temperature, catalyst and pressure to H_2O proportions are all variable in the HTG procedure, depending on the necessity for product assembling.

An increased output of organic garbage in current decades has put significant strain on the atmosphere and trash managing procedures. Among the biodegradable organic wastes that cause significant discomfort are waste paper, animal waste, garden waste, food waste, sludge and sewage (Heidari *et al.*, 2018). The major components of

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paper and biodegradable wastes are starch, cellulose, lignin and hemicellulose. Leading to a shortage of suitable ways for make use of such garbage, or rather a lack of study on the subject, they are either incinerated tor deposited into landfill sites, inflicting ecological degradation in the procedure. Waste dumped into land leads to air, water and land consumes valuable area, emits greenhouse gases into the surroundings, and contaminates freshwater. Incineration, frequently produces subordinate contaminants along with emission of carbon dioxide (Karthikeyan *et al.*, 2018). Energy from trash via char has gotten a lot of interest in the linear economy because of its energy security and energy restoration. Hydrothermal carbonisation (HTC) is used to transform organic material into solid fuel under mild circumstances (250°C, 60 bar).

In order to boost the CO + H_2 production, either more biomass or the inclusion of a suitable catalytic agent was selected. Metal-bound catalysts are proved as effective in producing H_2 from a variety of biomass. On the existence of a Ni-based catalyst, polythene trash was gasified with timber dust. The synergistic reactions in the system resulted in 37 % H_2 production when the aforementioned biomass combination was gasified with a Ni/Al_2O_3 catalyst. At the right work environment, like duration, solvent to biomass ratio and temperature, catalysts aid in the better decomposition of biomass or waste (Fozer *et al.*, 2019). Catalysts must be stable at both supercritical and subcritical temperatures. For the program to be successful, it was also necessary to build a low-cost, effective, recyclable, and abiding catalyst. Clay-based catalysts will be an economical assistance in the development of improved catalysts.

The declaration of originality highlights the important energy banks (Domestic waste) for H_2 energy generation. This research proposes a long-term solution for trash reduction and energy generation. In this study, the optimisation of bio-oil upgradation was performed, as this paves way for further research for effective usage of domestic waste. In the earlier days, there were limited research dedicated to the manufacture of green fuels from home combined garbage in the current generation. Domestic trash were employed as fuel for H_2 generation using the hydrothermal gasification and liquefied method in this study.

2. Resources and approaches

2.1. Domestic waste separation and study

The waste biomass for our investigation was gathered from diverse domestic waste. Food waste, organic trash (paper), and plastic trash were all mixed together (Hietala *et al.*, 2019). The generated trash mixture was chopped down to a regular size of 2 mm using a blade shredder. Wet and evaporative content of waste was employed to assess the trash's proximate configuration. The fundamental configuration of the trash was set on employing a CHNS analyser (Mahssin *et al.*, 2021). The degrading gradient of trash in relation to temperature was investigated using a Shimadzu TGA 60 H Analyser and thermo-gravimetric measurement (Sztancs *et al.*, 2020).

2.2. Hydrothermal gasification experiments

Mixed waste HTG tests on H_2 yield were carried out in a Parr reactor with a capacity of 260 millilitre and an electric warming and refrigeration device (Jayaraman *et al.*, 2022). Before the main tests, the apparatus was warmed at 450°C for 5 h with H_2O acting as a solvent to remove any remains. Inside the reactor, a nitrogen-rich atmosphere was sustained. For 1 h, the impact of water to ethanol ratios, solvent to waste ratios, temperature (290–430°C), catalyst load (2-4 wt.%), and pressure (6 MPa) on HTG were investigated (Arregi *et al.*, 2017). Bentonite/Nb-TiO_2 catalyst was made using the sol-gel process involving very minor modifications. After the gasification tests were completed, the reactor was chilled to extract the char, gas, and liquid phases.

2.3. Setup for hydrothermal liquefaction

At a N_2 atmosphere of 6 MPa and reaction duration of 1 hour, HTL studies were primarily used to extract bio-oil and prized gases. On the HTL of waste, the effects of catalyst level (2–6% wt.), temperature (300–400°C), and solvent to waste proportion were investigated. Upon disassembling the reactor, volatile components were retrieved through exhaust (Jayaraman *et al.*, 2022). Bio-oil was removed from the dark brown crude generated following HTL trials using dichloromethane. To separate the phases, an equal amount of DCM was put into the bio-crude, stirred, and put into a separatory funnel. Equations were used to compute the bio-oil production and higher heating value (HHV) rate (1 and 2).

$$\text{Bio-oil yield (weight percentage)} = \text{Mass of bio-oil yield (grams)} / \text{Mass of bio-mass (grams)} \times 100 \quad (1)$$

$$\text{HHV (MegaJoule/kilogram)} = 0.44 * C + 1.62 * (H-O/7.9) \quad (2)$$

2.4. Study of HTG and HTL outcomes

The gaseous product proportion produced from both HTG and HTL trials was examined using gas chromatography (GC). The gaseous product was examined employing a gas chromatograph (GC) along with a thermal conductivity detector (TCD). The stream velocity of carrier Helium gas was 30 millilitre/minute (Su *et al.*, 2020; Nguyen *et al.*, 2020). The oven's initial temperature was kept at 55°C for one minute before being increased to a temperature of 120°C at a percentage of 25°C /minute. For 5 min, temperature was held constant. FID detectors in an Agilent 7900 GC were used to typify the bio-oil. The content of bio-oil was determined according to the proximal and final compositions of the by-products were determined.

2.5. Enhancement studies by response surface methodology (RSM)

The optimal factors on bio-oil outcome through liquefaction and H_2 gas outcome through gasification were identified using a central composite design (CCD) with third stage third component assessments (Khoo *et al.*, 2020). The 3 independent variables are temperature (260–450°C), catalyst level (2-4%), and solvent to waste (20–60). The production of bio-oil and H_2 was measured in wt.%. Eq.

states the association amid variables and reaction (Lecker *et al.*, 2017).

$$Y = \beta + \sum_{i=1}^{N} \beta i \times Xi + \sum_{i=1}^{N} \beta ii \times Xi + \sum_{i=1}^{N} \sum_{j=i=1}^{N} \beta ij \times Xii$$

3. Result and discussions

3.1. Features of municipal waste

Thermal deprivation of trash was investigated in accordance with the literature in order to evaluate thermostability and the best thermal gradient for gasification trials. The TGA profile of home waste is shown in Figure 1. The loss in weight was exactly linked to the temperature series of 350–450 °C, according to the findings (Siwal *et al.*, 2020). At 800°C, a maximum weight loss of 80% was reported at a heating frequency of 12 degree Celsius /minute. Carbon accounted to 57 percent of the trash, followed by Oxygen at 31%, Hydrogen at 8%, Nitrogen at 7.03%, and Sulphur at 1.37%, according to the findings of the elemental composition. In gasification studies, the waste's low moisture (11.3%) and ash (11.8%) levels were advantageous (Arun *et al.*, 2020). The waste's HHV value was discovered to be 24.8 MJ/kg. Food waste included 42%, 7.5%, 13%, 1%, and 34% carbon, sulphur, hydrogen, oxygen, sulphur, and nitrogen, according to recent research.

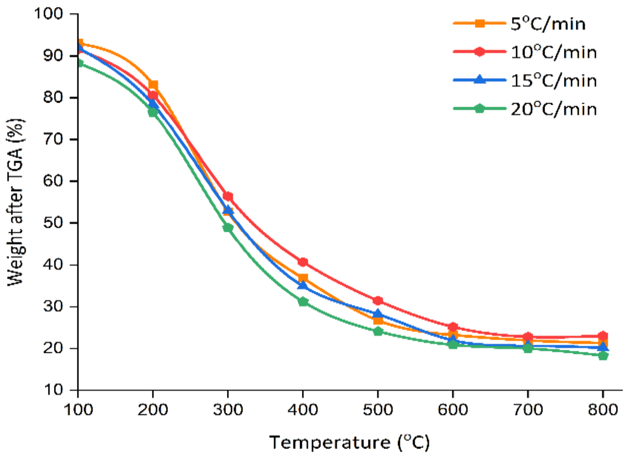

Figure 1. TGA graphical depiction of domestic waste

3.2. Hydrothermal gasification

3.2.1. Influence of temperature on product distribution

The reaction temperature is significant because it disturbs the configuration of the products and the H_2 production (Oh *et al.*, 2018). Temperature has an important influence on product output, according to research, since enthalpies, particularly those of H_2 and CH_4, were associated with development factors. It has been also claimed that in the HTG process, quicker heating rates are required to prevent downsides such as reactor blocking (Choiron *et al.*, 2020). Considering all of this in mind, the HTG process was kept at a temperature varying from 290 - 450 °C. The temperature and gas production are exactly related, as seen in Figure 2. A reaction temperature of 400-degree Celsius payed way for a larger percentage of gas product production (40.7 weight percentage), with solid remnant and liquid goods respectively at 9 and 54 weight percentage.

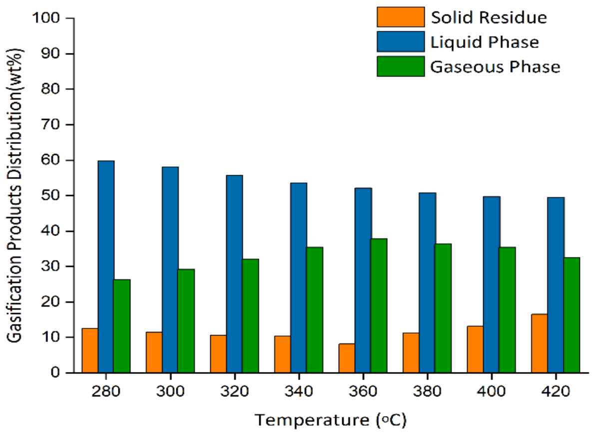

Figure 2. Impact of Hydrothermal Gasification product segregation

3.2.2. Influence of solvent

Conferring to the results, a suitable and process solvent should be employed to generate improved H_2 by gasification. The influence of water and ethanol in different ratios on H_2 production was examined in this work (Nanda *et al.*, 2015). When H_2O and C_2H_5OH was employed in equivalent amounts in the HTG trials (Figure 3), the maximal H_2 gas output was 35 wt.% (1:1). Inside the mixture of sum of gaseous products acquired in the procedure, this H_2 outcome was calculated (Hietala *et al.*, 2019). The increased hydrogen generation was attributable to the solvent system's accelerated waste breakdown and energy transmission at a 2:2 ratio. In the presence of ethanol, waste biomass gasification resulted in a more approving heat and mass transfer environment (Kumar *et al.*, 2020).

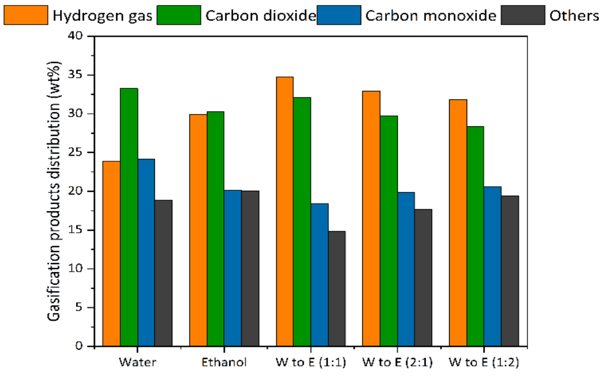

Figure 3. Impact of solvent on hydrothermal gasification gaseous product segregation.

3.2.3. Influence of solvent to waste ratio

For a noteworthy concentration of waste gasification and H_2 production, an ideal solvent to waste proportion was found to be acute. It was stated that a lack of solvent might cause problems and a reduced productivity (Jayaraman *et al.*, 2022). As a result, the optimal solvent load in the HTG process has to be determined. HTG studies were carried out with varied solvent to waste ratios of 50, 30, 14, and

15 mL/g in order to develop the perfect mixture of solvent to waste proportion (Dimitriadis and Bezergianni, 2017). The contribution of solvent to trash in the gasification method was examined and depicted in Figure 4. For 14, 15, 30, and 50 mL/g, the hydrogen production was 32, 29.0, 27.2, and 23.1 wt.%, correspondingly. During the gasification process, processes such methanation, water gas shift and reforming were credited with highest hydrogen output.

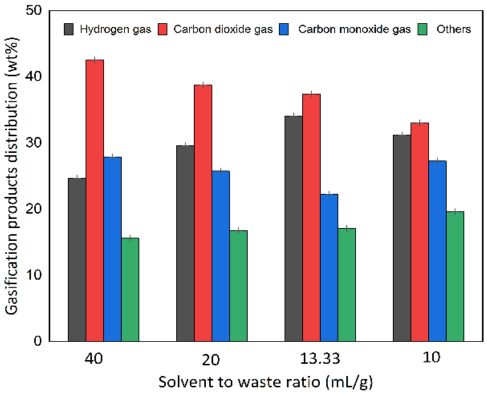

Figure 4. Impact of solvent to bio-mass proportion on gasification procedure.

3.2.4. *Impact of catalyst*

The catalyst level must be enhanced since it determines the fraction of product outcome. The sol-gel technique produces uniformly sized particles, according to the well-defined study of the bentonite/Nb-TiO_2 catalyst. The area of the catalyst was 38 metre^2/gram when 6 weight percentage Niobium was laden in a 15 weight percentage bentonite catalyst, conferring to Brunauer–Emmett–Teller (BET) study. The pore volume was 6 nm, indicating that adding Nb to TiO_2 inhibited crystal development (Arregi *et al.*, 2017). The HTG studies were put in 5 distinct reaction catalyst loading (2-6% wt. %), and the influence on gaseous product outcome is depicted in Figure 5. The gas development pattern was comparable to reaction temperature, with the highest gas formation at 43 wt.% and the lowest at 5 wt.%. It included 41% hydrogen gas, 23% CO_2 gas, and 21% CO gas, correspondingly. Inside the HTG reactor, methanation, water gas shift and hydrolysis processes ended in a larger volume of gas product generation (Siwal *et al.*, 2020). The inclusion of a catalyst to the reaction phase resulted in increased bio-mass gasification and decreased char development. It demonstrates that catalysts would aid in energy conservation and simplify usage. At increasing catalyst concentrations and temperatures, chemicals with low molecular weight in the liquid phase are transformed to gas molecules. The reaction's liquid result can be treated to the steam reforming method to produce more biofuels. The gasification of *S. obliquus* biomass on the existence with 4 weight percentage consumed Calcium hydroxide produced 38 wt.% H_2 gas.

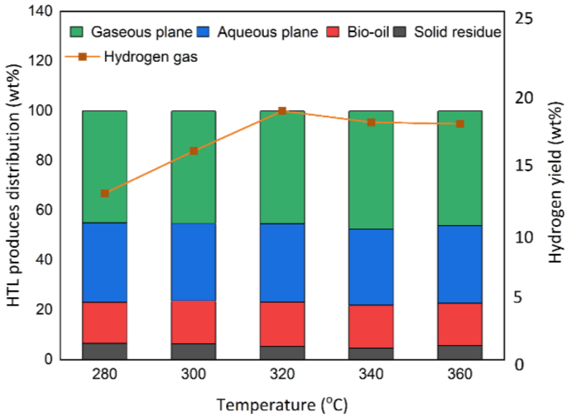

Figure 5. Part of Catalyst in HTG process (Solvent to waste proportion: 14.2 millilitre/gram, solvent H2O to C2H5OH (2:2), Temperature: 400°C)

3.3. *Hydorthermal liquefaction*

3.3.1. *Influence of temperature in HTL process*

The HTL trials for biofuel generation were put in at temperatures reaching 290 to 400 °C for 1 h. The maximal bio-oil generation was 30 wt.%, the solid remains was 11 wt.%, and the aqueous phase was 30 wt.%, according to the results. At 360°C, the H_2 gas outcome was 19 wt.%, and the CO_2 gas yield was 31.0 wt.% in the gas product (33 wt. %). Because there was inadequate solvent and waste breakdown at lesser temperatures, development of solid remnants was more. Temperature was shown to be inversely related to the amount of bio-oil when hydrocarbons were disintegrated. With a larger fraction of gaseous generation, the hydrocarbon output was lowered. Without a catalyst, municipal solid waste yielded 56.4 % biocrude at a temperature of 550°C. Temperature is the key elements that determines the standard of bio-oil output, according to numerous research. Increases in temperature over the ideal threshold, on the other hand, resulted in lower biocrude yields.

3.3.2. *Impact of solvent to waste proportion*

Figure 6 shows the bio-oil and H_2 yields in the Hydrothermal liquefaction procedure for different solvent to trash ratios (50, 30, 14, and 15 mL/g). The analysis indicated that the solvent to waste proportion had a noteworthy impact on the production of bio-oil and hydrogen. At 360 °C and a solvent to weight proportion of 14 mL/g, the HTL procedure yielded bio-oil of 30.4 weight percentage, solid remains of 12 weight percentage, gas of 33.2 weight percentage, and aqueous phase of 29 weight percentage. 21.7 weight percentage hydrogen gas, 31.7 weight percentage Carbon-dioxide gas, and 25.5 weight percentage Carbon-monoxide gas was included in the 41.1 weight percentage gas product, correspondingly. The fraction of gaseous products formed increased as the solvent to waste proportion increased. The formation of more gas was instigated by the interruption of evaporative chemicals in liquid properties. When ethanol was utilized in the same quantity as water, it caused more breakdown of biomass compounds. This is because ethanol produces active H+, which hinders the repolymerization step.

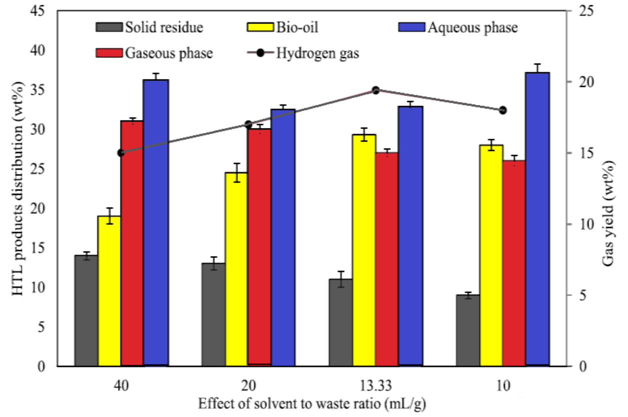

Figure 6. Graphical representation of Impact of Solvent - waste proportion in hydrothermal liquefaction process

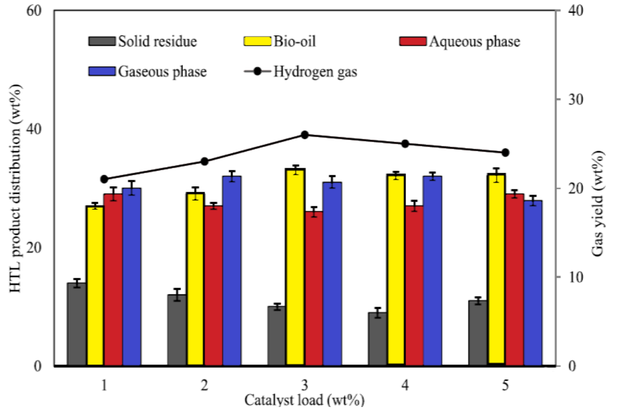

Figure 7. Graphical representation of impact of catalyst on HTL process

3.3.3. Impact of bentonite/Nb-TiO₂ catalyst on hydrothermal liquefaction method

The part of bentonite/Nb-TiO_2 in liquefaction-based concurrent bio-oil and H_2 generation was investigated. Figure 7 depicts the product distribution and hydrogen production in the HTL process. At a temperature of 360 degree Celsius, a catalyst load of 4 weight percentage, and a solvent to proportion of 14 millilitre/gram, the solid remains, gas generation, aqueous phase and bio-oil were 15, 30, 39, and 29 wt.%, correspondingly. The gas outcome had 28% H_2 gas, 25.5% CO_2, and 25% CO, correspondingly. The resulting bio-oil contained a carbon content of 84 wt.% and an oxygen level of 9.4 wt.%, according to the final analysis. The calorific value of the liquefied bio-oil was 35.6 MJ/kg, with Hydrogen by Carbon and Oxygen by Carbon ratios of 1.2 & 1.3, correspondingly. The usage of a subcritical state at the time of this process assistances in the formation of more bio-oil via hydrogenation processes (Bej *et al.*, 2014). Due to the availability of substrates via deoxygenation, reduction, and depolymerization procedures, the frequency of methanation, water gas shift, and reforming processes increases. The HTL method may be enhanced further by using solar energy for warming and employing catalysts that are selective to bio-mass deprivation in proportion to intended product generation. The catalyst's role in the HTL method was to reduce the likelihood of biochar production by boosting biocrude output. It should also reduce the synthesis of intermediate molecules by processes like condensation and repolymerization.

3.4. Optimization of HTL and HTG processes by RSM

The RSM experiment was conducted to determine the optimal temperature, solvent to waste and catalyst level proportion for producing bio-oil and H_2 in the liquefaction and gasification processes, correspondingly. Temperatures of 300–456 °C, catalyst concentrations of 2-6%, and solvent to trash weight ratios of 20–60 mL/g are among the factors employed in RSM (Paida *et al.*, 2019). With 30 runs, CCD tests for both the HTL and HTG processes were examined to determine the best parameters for bio-oil and hydrogen output. The statistical dispersion of ANOVA test is detailed in Table 1. When two parameters are evaluated, the findings show that solvent - trash and temperature are the most crucial elements for bio-oil production in the HTL procedure. Nevertheless, catalyst load and temperature are the most important elements in determining hydrogen output solvent to effluent. ANOVA table showed the co-efficient of regression value for the two processes. The experimental results matched in the HTG and HTL processes had R^2 values of 1.0 and 1.20, respectively (Figures 8 and 9). The expected R^2 values were 1.76 and 1.56, correspondingly, which matched the corrected R^2 values of 1.25 and 1.28 for the HTG and HTL processes. As a result, the empirical formula was rewritten as follows:

Bio-oil yield (wt. %) = − 49.10 + 0.40 Temperature + 2.76 catalyst load + 0.48 (Solvent to trash) − 0.1 (Temperature) 1.8 − 0.20 (catalyst wt.)1.8 − 0.1 (Solvent to waste) 1.8 − 0.1 (Catalyst load ×Temperature) − 0.1 (Catalyst load × Solvent to waste) + 0.1 (Temperature × Solvent to waste)

Hydrogen yield (wt. %) = – 20.73+0.20 Temperature – 2.01 catalyst load + 1.02 (Solvent to trash) – 0.01 (Temperature) 1.8 – 0.30 (catalyst wt.) 1.8 – 0.1 (Solvent to waste) 1.8 + 0.1 (Catalyst load × Temperature) – 0.1 (Catalyst load × Solvent to waste) + 0.1 (Temperature× Solvent to waste)

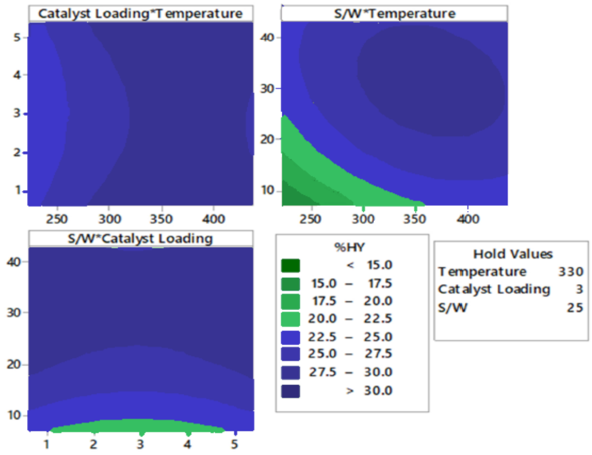

Figure 8. Graphical representation of outline subversions for H_2 generation in gasification methods.

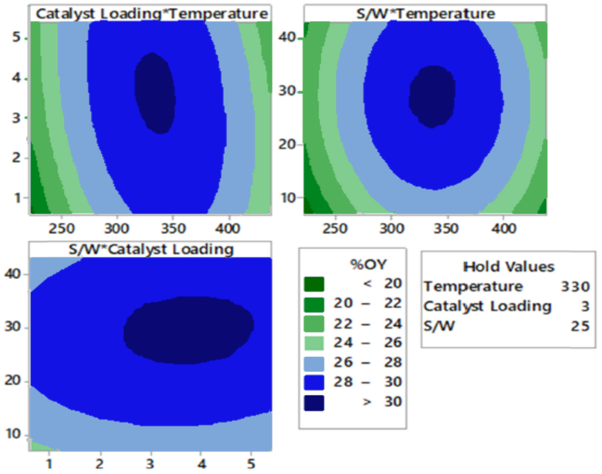

Figure 9. Graphical representation of outline plots for the bio-oil generation in liquefaction methods.

3.5. Overall H_2 capability of domestic waste

Waste - energy conversion is required in today's society as a means of reducing waste and meeting energy demands. The bio-energy capacity of domestic garbage was assessed using the HTG and HTL processes in this study. The HTG method is essentially focused with the generation of bio-hydrogen, whereas the HTL method is mainly concerned with the creation of bio-oil. Nevertheless, as a by-product of the HTL procedure, bio- H_2 was produced. As a result, the total H_2 capability of domestic garbage was calculated using both processes. The highest H_2 production from the HTG method was 40 wt.%. The bio- H_2 production from the hydrothermal liquefaction process was 27 weight percentage, but also produced bio-oil with an HHV of 39 Mega Joule/kilogram. In the presence of appropriate photo catalysts, the aqueous phase generated from both processes can be regenerated (Alvarez *et al.*, 2014). This will increase the total bio-energy efficacy of domestic waste by a significant amount of hydrogen. It was clear from this investigation that home garbage is a useful reserve for the manufacture of not so dangerous items. These products will be used as an alternative to fossil fuels. Because these products are mostly regarded green fuels with a carbon cycle that is carbon neutral, they add less to greenhouse gas emissions and climate changing actions (Sztancs *et al.*, 2020).

4. Conclusion

In this work, domestic mixed trash was treated in C_2H_5OH to H_2O solvent system at temperatures ranging from 290 to 440 °C (gasification) and 290 to 400 °C (liquefaction) in the existence and absence of bentonite/Nb-TiO_2. FT-IR, GC-MS, and fundamental study were used to examine the biochemical characteristics of liquid hydrocarbons produced by the HTL procedure. With a calorific value of 35 Mega Joule/kilogram and Oxygen by Carbon and Hydrogen by Carbon ratios of 1.125 & 1.0, liquid fuel formation was 34 wt.%. The biohydrogen composition is determined by GC analysis of the gaseous products from both procedures. For a solvent to waste proportion of 14 millilitre/gram, C_2H_5OH to H_2O of 2:2, and a duration of 1 hour, the H_2 output from hydrothermal liquefaction was 28 weight percentage (catalyst loads: 4 wt %) and Hydrothermal gasification was 40 wt % (catalyst loads: 5 wt%). Temperature and the solvent-trash proportion were the ideal factors in H_2 synthesis methods, according to optimization analysis. According to the findings of this review, home garbage will be a viable supply for bio-energy creation. In the forthcoming years, there will be a need to identify prolonged storage of bio-hydrogen. This study will act as a base for future studies into the extraction of bioenergy from domestic garbage, with a focus on stowage and transportation options.

Conflicts of interest

The authors declare that there is no conflict of interest.

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