Pretreatment of Korean pine (*Pinus koraiensis*) via wet torrefaction in inert and oxidative atmospheres

Quyen Nguyen[a,#], Dinh Duc Nguyen[b,c,#], Chao He[d] and Quang-Vu Bach[e,*]

[a]*Institute of Research and Development, Duy Tan University, Danang 550000, Vietnam*

[b]*Faculty of Environmental and Food Engineering, Nguyen Tat Thanh University, 300A Nguyen Tat Thanh, District 4, Ho Chi Minh City, 755414, Vietnam*

[c]*Department of Environmental Energy Engineering, Kyonggi University, Suwon 442-760, Republic of Korea.*

[d] *Faculty of Engineering and Natural Sciences, Tampere University, Tampere, Finland*

[e]*Sustainable Management of Natural Resources and Environment Research Group, Faculty of Environment and Labour Safety, Ton Duc Thang University, Ho Chi Minh City, Vietnam*

[#] These authors contributed equally to this work

* Corresponding author at Faculty of Environment and Labour Safety, Ton Duc Thang University, Ho Chi Minh City, Vietnam.

E-mail: bachquangvu@tdtu.edu.vn

Abstract

This study investigates the possibility of utilizing waste heat sources such as hot flue gas or hot air for wet torrefaction (WT) processes. Although waste heat sources are cheaper alternatives than pure nitrogen used as purging and pressurizing gas for WT, they contain some fractions of non-inert gases and potentially affect the yield and fuel properties of the solid product (hydrochar). To assess these possible influences, Korean pine (*Pinus koraiensis*) was subjected to WT in different atmospheres, including N_2, CO_2, O_2, air and synthetic flue gas (SFG), and the produced hydrochars were characterized. The results show that WT in different gas atmospheres at 200 °C and 20 bar yields 72.24–73.82% of hydrochar. In general, the fuel properties of the hydrochars are found to be superior to those of the untreated pine: the O/C ratio decreases from 0.703 (raw pine) to 0.582–0.588 (hydrochars). Furthermore, analysis of variance (ANOVA) and pairwise t-test were performed, and the results reveal that the effects of different atmospheres in WT are not statistically meaningful (p-values > 0.05), except for the influence of CO_2 on the ash content. The results also indicate that the presence of oxidative gases such as O_2 and CO_2 in the WT process at appropriate pressures and temperatures has almost insignificant effects on the yields and fuel properties of the hydrochar products. The findings from this study could encourage the utilization of waste heat sources for WT to reduce operating costs.

Keyword: Wet torrefaction; Hydrochar; Biochar; Fuel properties; Waste heat; Oxidative gas.

1 Introduction

Wet torrefaction (WT) is a hydrothermal pretreatment of biomass at temperatures of 180–260 °C [1], and can be performed under saturated or elevated pressures. WT is very suitable for wet biomass such as agricultural residues, sewage sludge, and aquatic wastes, which have relatively high moisture contents. The main product from WT is hydrochar, a solid fuel with better fuel properties, more hydrophobicity, and better grindability than untreated biomass [2-10]. Remarkably, hydrochar has a lower O/C ratio and thus better a heating value than raw biomass. Moreover, the combustion reactivity of hydrochar is improved after WT [11]. In addition, hydrochar also has a lower ash content than native fuel, which is beneficial for further thermal conversion systems regarding ash-related issues.

Another process producing solid fuel similar to WT is dry torrefaction (DT), which is defined as a thermal pretreatment of biomass without the presence of oxygen at 200–300 °C [12]. The main solid product from DT is called biochar or dry-torrefied biomass. While WT is normally conducted in a pressurized reactor and employs hot liquid water as the reaction medium; DT is operated at atmospheric pressure and under a stream of inert gas (nitrogen or argon). Because water is a more reactive medium than inert gas, WT requires a lower temperature than DT to produce the same amount of solid product [2]. In addition, hydrochar from WT has higher energy content than biochar from DT obtained under comparable conditions that produce the same solid yield. Moreover, the heating value of hydrochar is comparable to lignite and sub-bituminous coals [1]. As an energy-dense material, hydrochar is ideally utilized for energy applications; it can be burned directly as a solid fuel to produce heat and power, or it can be converted into liquid or gaseous fuels via subsequent thermochemical conversion processes (e.g., pyrolysis, liquefaction, and gasification). Furthermore, hydrochar and its derivatives can be potentially employed as soil amendments and adsorbent materials [13].

Currently, most WT studies at the laboratory scale are conducted in batch reactors, in which biomass and water are mixed at desired ratios. Thereafter, an inert gas (e.g., nitrogen) is normally used to purge and/or pressurize the reactor [1]. For industrial applications and up-scaling of WT technology, the required amount of inert gas increases significantly and thus becomes costly. A potential option to address this issue is the utilization of waste heat sources such as hot flue gas or hot air, from which the available heat can be integrated for both heating and pressurizing the WT system, considering that WT is operated at 180–260 °C. Successful integration not only reduces the cost of inert gas but also decreases the energy consumption to heat the system. However, these waste heat sources always contain other components such as CO_2 and O_2 at different concentrations, which may affect the product yield and fuel properties of the produced hydrochar, due to their oxidative reactivities. Nevertheless, no WT study was found in the literature to address these issues, except a few works employing CO_2 in hydrothermal media to utilize its acidic catalytic effect [14-17].

The utilization of non-inert gas as a substitute for nitrogen has been actively researched for DT process. Instead of N_2, several non-inert gases and gas mixtures have been employed for DT, including CO_2 [18-21], partial oxygen [22-24], air [25, 26] and simulated flue gas (SFG) [20, 27]. These studies found that the presence of non-inert gases such as CO_2 and O_2 in DT significantly reduced the biochar yield. In addition, the biochars produced by DT in non-inert atmospheres had inferior fuel properties to those produced by inert atmospheres. These unwanted effects are due to oxidation reactions between the biomass feedstock and the oxidative gases [22]. Therefore, optimization of DT conditions and controlling the oxidative gas concentration in DT gases have been intensively studied to mitigate the negative effects of non-inert gases. Nevertheless, WT under oxidative atmospheres has not been paid enough attention; thus, the effects of oxidative gases on the yield and fuel properties of the hydrochar remain unclear. Therefore, it is necessary to investigate oxidative WT to determine whether the effects of oxidative gases on WT are similar to those on DT.

For the aforementioned purpose, WT of Korean pine (*Pinus koraiensis*) was performed in a batch reactor in several atmospheres including N_2, CO_2, O_2, air, and simulated flue gas (SFG). The yield and fuel properties of the hydrochars produced in different atmospheres were analyzed and compared. Among the atmospheres, N_2 served as the base case; pure O_2 and air (79 vol% N_2 and 21 vol% O_2) were employed to study the effect of oxygen, while pure CO_2 and SFG, which contains 90 vol% CO_2 and 10 vol% O_2, were chosen to examine the role of CO_2. It should be noted that inert gas (i.e., N_2) was excluded from the SFG to observe a clearer effect of the non-inert gases. A real flue gas exhaust may contain a large amount of N_2 (in traditional power plants) or no N_2 (in modern power plants with oxy-fuel combustion). Comparisons of the yield, proximate and ultimate analyses as well as the energy content of the hydrochars produced in different atmospheres are presented. In addition, analysis of variance (ANOVA) and pairwise t-test were performed to determine if the differences in the yields and fuel properties of the hydrochars due to the changes in the WT atmosphere are statistically significant.

2 Experimental methods

2.1 Feedstock and hydrochar characterization

Korean pine (*Pinus koraiensis*) was purchased from a local supplier and ground to have particle sizes of 3–5 mm. It was then dried at 105 °C for 24 h and stored for further use. The proximate analyses of both the dry feedstock and hydrochar were performed according to the ASTM standards E871, E872, and D1102 for the moisture, volatile matter, and ash contents, respectively. The ultimate analysis was conducted using a EuroVector Euro EA Elemental Analyzer. The heating value was calculated from the correlation proposed by [28]. The main fuel properties and the heating value of the feedstock are listed in **Table 1**.

Table 1. *Main fuel properties and heating value of Korean pine (Pinus koraiensis)*

Characteristics	**Unit**	**Value**
Proximate analysis		
Moisture	*wt%, ar*	9.63
Volatile Matter	*wt%, db*	86.72
Fixed Carbon	*wt%, db*	11.85
Ash	*wt%, db*	1.43
Ultimate analysis		
Carbon	*wt%, daf*	48.28
Hydrogen	*wt%, daf*	5.983
Nitrogen	*wt%, daf*	0.511
Oxygen (by difference)	*wt%, daf*	45.23
Lower heating value (LHV)	*MJ/kg, daf*	17.90

ar: as received,
db: dry basis,
daf: dry and ash-free

2.2 Wet torrefaction

WT experiments were conducted in a 1 litter batch reactor, which was designed and built for hydrothermal conversion applications at Kyonggi University. The system, illustrated in **Figure 1**, consists of a stainless steel reactor, pressure gauge, stirrer, pressurized vessels, pressure-reducing valve, heating element, hydraulic cooling system, thermometer, and control unit. More details of the system can be found in [29]. For each WT run, approximately 50 g of dry biomass and 250 g of distilled water were loaded into the reactor, which ensured that the mass ratio of biomass and water was 1:5. Different gases and gas mixtures (including N_2, CO_2, O_2, air, and SFG) were used to purge and pressurize the reactor. Thereafter, the reactor was heated to a WT temperature of 200 °C, at which the reactor pressure increased to approximately 20 bar. The system was maintained at this temperature for 30 min, counted as residence time, and then cooled with tap water. The gaseous products were not collected in this study, while the solid and aqueous products were separated using filter paper (Whatman grade 4). The final solid (hydrochar) was dried at 105 °C for 24 h and stored in a desiccator prior to further analyses.

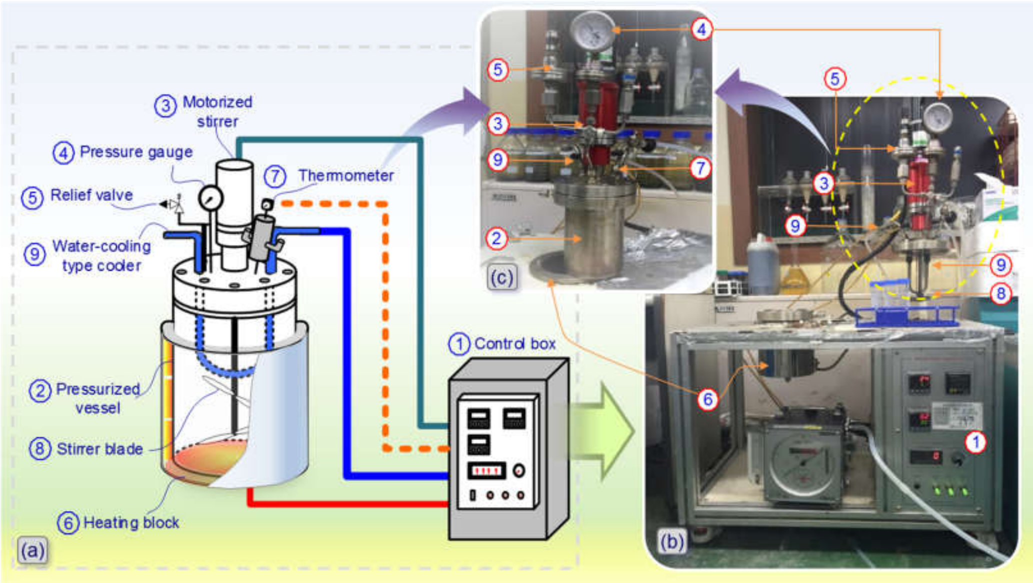

Figure 1. *Wet torrefaction system: schematic diagram (a) and actual photographs (b, c).*

2.3 Analysis of variance (ANOVA) and pairwise t-test

In this work, one-way ANOVA was employed to evaluate whether WT in different atmospheres (i.e., groups) had a clear effect (statistical significance) on the yield and fuel properties of the produced hydrochars (i.e., variances). Furthermore, a pairwise t-test was employed to determine which pair of groups differed from the others. These tests were performed at a significance level of 0.05 and a null hypothesis was proposed that there is no difference (i.e., no effect on the hydrochar yield and fuel properties) among the atmospheres. If the one-way ANOVA gives a p-value less than 0.05, it is against the null hypothesis, which indicates that at least two atmospheres have significantly different effects. Otherwise, unpronounced differences can be concluded if the p-value is greater than 0.05. Similarly, if a pairwise t-test for any two atmospheres results in a p-value < 0.05, it means that the effects between the two atmospheres are statistically meaningful and vice versa.

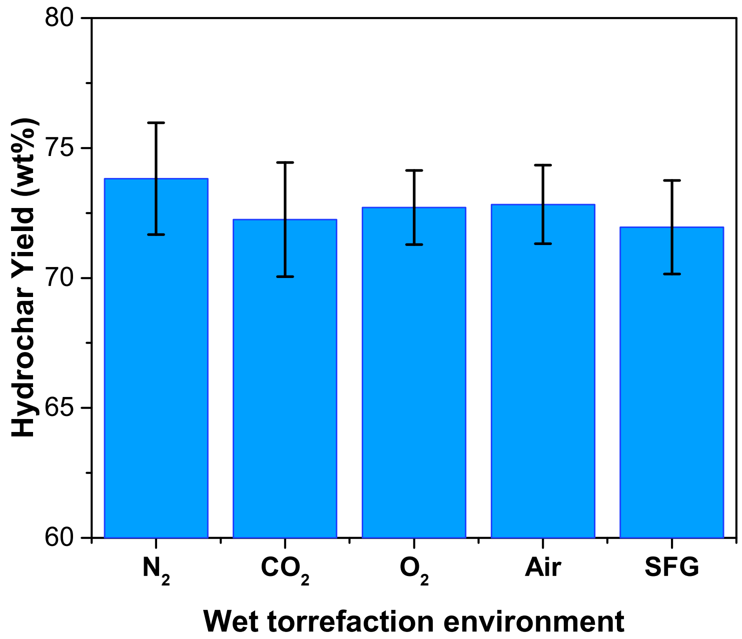

Figure 2. *Hydrochar yield from WT of Korean pine in different atmospheres*

3 Results

3.1 Solid yield

Solid yields of the hydrochar produced from WT at 200 °C and 20 bar in different atmospheres (including N_2, CO_2, O_2, air, and SFG) are presented in **Figure 2**. Among the atmospheres, WT in N_2 yielded the highest hydrochar content (73.82%), while WT in CO_2 produced the lowest amount of hydrochar (72.24%), and WT in other atmospheres yielded hydrochar contents in between the lowest and highest (72.71% for O_2, 72.81% for air and 72.95% for SFG). Therefore, ANOVA was performed to confirm whether these differences are statistically significant. The test gave a p-value of 0.7710 (see **Appendix A1**), which reveals that WT atmosphere has an insignificant effect on the hydrochar yield. This observation is different from DT, in which torrefaction gas plays an important role. When N_2 was replaced by air or CO_2, the biochar yield dramatically reduced because of the effect of the oxidative atmospheres.

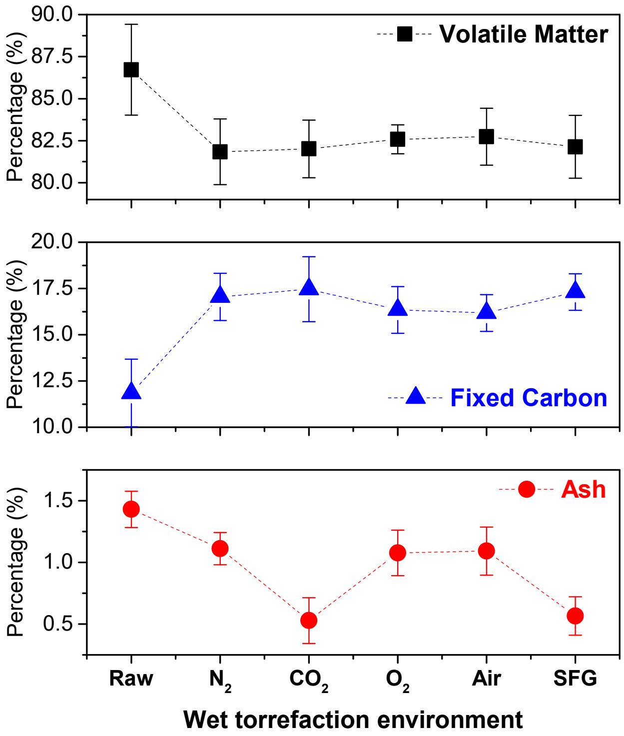

Figure 3. *Proximate analysis of raw Korean pine and its hydrochars obtained from WT in different atmospheres*

3.2 Proximate analysis

Results from the proximate analysis of hydrochars obtained from WT in different gases at 200 °C and 20 bar are demonstrated in **Figure 3**. At the first sight, it can be observed that the ash content in the hydrochars from WT in CO_2 and SFG were lower than the others. The ANOVA gave a p-value of 0.0023 (see **Appendix A1**), which confirms that there are significant differences among the WT atmospheres with regard to the ash contents of the produced hydrochars. Hence, pairwise t-tests were conducted, and the results from these tests are illustrated in **Figure 4**. It is evident from the figure that the ash contents in hydrochars from WT in CO_2 and SFG were different from the others (p-value < 0.05); however, the effects of the two atmospheres do not differ from each other ($p = 0.8054$). The reason for this observation is the role of CO_2, which potentially has an acidic catalytic effect in hydrothermal media and thus can dissolve part of the ash elements.

On the other hand, **Figure 3** reveals that the volatile matter contents of the hydrochars decreased while their fixed carbon contents increased after WT. The volatile matter decreased from 86.72% in the raw biomass to 81.80–82.73% in the hydrochars, while the fixed carbon increased from 11.85% in the raw biomass to 16.17–17.46% in the hydrochars. In addition, the ANOVA showed p-values of 0.9551 and 0.6679 for the volatile matter and fixed carbon contents, respectively (see **Appendix A1**). These results indicate that the WT atmosphere has unpronounced effects on both the volatile matter and fixed carbon contents, even though it influences the ash content. This is because the ash content in the raw pine is relatively low (only 1.43%); thus, a significant change in ash content could not cause pronounced differences in the volatile matter or fixed carbon contents.

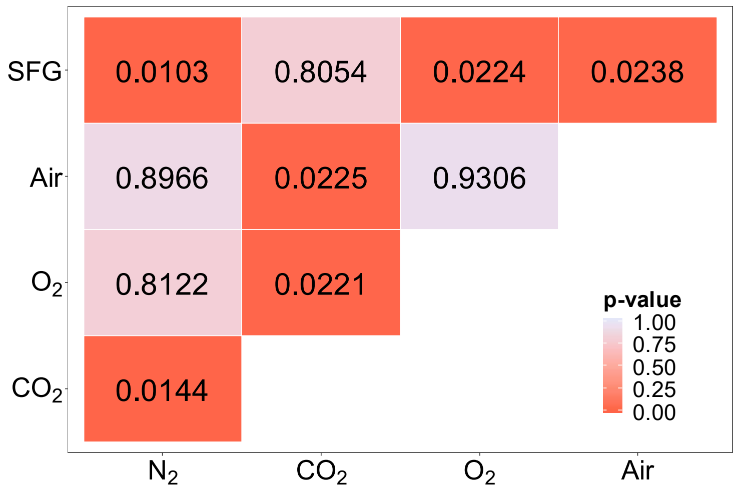

Figure 4. *Illustration of p-value from pairwise t-tests for ash contents of hydrochars*

3.3 Ultimate analysis

The major elemental compositions through ultimate analyses of the hydrochars produced by WT at 200 °C and 20 bar in different atmospheres are presented in **Table 2**. The table shows typical changes in the elemental composition of hydrochars after WT, such as increases in the carbon and nitrogen contents, and decreases in the hydrogen and oxygen contents. In detail, the carbon content increased from 48.28% in raw biomass to 52.63–52.76%; the hydrogen content decreased from

5.983% in raw biomass to 5.697–5.779%; the nitrogen content increased from 0.511% in raw biomass to 0.539–0.549%, and the oxygen content decreased from 45.23% in raw biomass to 40.91–41.19%. Consequently, the O/C ratios of the hydrochars reduced from 0.703 to 0.582–0.588, while their H/C ratios decreased from 1.487 to 1.300–1.314 after WT. Nevertheless, the influence of the WT atmosphere on the elemental composition of hydrochars was insignificant, which is reflected by the ANOVA p-values. The p-values for the carbon, hydrogen, nitrogen, and oxygen contents were respectively 0.9997, 0.9350, 0.9417, and 0.9991 (see **Appendix A1**).

Table 2. *Ultimate analysis of hydrochars produced by WT in different atmospheres (on dry and ash-free basis)*

	C (*wt%*)	**H** (*wt%*)	**N** (*wt%*)	**O** (*wt%*)	**O/C ratio**	**H/C ratio**
Raw	48.28 ± 1.50	5.983 ± 0.142	0.511 ± 0.019	45.23 ± 1.58	0.703	1.487
N_2	52.73 ± 1.41	5.759 ± 0.072	0.549 ± 0.011	40.96 ± 1.34	0.583	1.311
CO_2	52.57 ± 2.11	5.697 ± 0.068	0.539 ± 0.016	41.19 ± 2.03	0.588	1.300
O_2	52.63 ± 1.47	5.751 ± 0.107	0.545 ± 0.010	41.07 ± 1.59	0.585	1.311
Air	52.76 ± 1.14	5.779 ± 0.215	0.546 ± 0.019	40.91 ± 1.06	0.582	1.314
SFG	52.55 ± 1.58	5.721 ± 0.116	0.543 ± 0.016	41.19 ± 1.66	0.588	1.306

3.4 Heating value and energy yield

Figure 5 demonstrates the heating value and energy yields of raw Korean pine and its hydrochars obtained from WT in different atmospheres at 200 °C and 20 bar. The figures show that the LHVs of the hydrochars were enhanced while the energy yields were reduced after WT, which is in good agreement with other WT studies [2, 5]. In **Figure 5**A, it seems that the LHVs of hydrochars were unaffected by the WT atmospheres. This was confirmed by the ANOVA for LHV, which gave a p-value of 0.9965 (See **Appendix A1**). On the other hand, the energy yields (**Figure 5**B) of hydrochars produced by WT in CO_2 and SFG (78.19% and 77.95%, respectively) were slightly lower than the others (80.54% for N_2, 79.07% for O_2, and 79.61% for air). However, the ANOVA for the energy yield resulted in a p-value of 0.8246, which implies that these differences are insignificant.

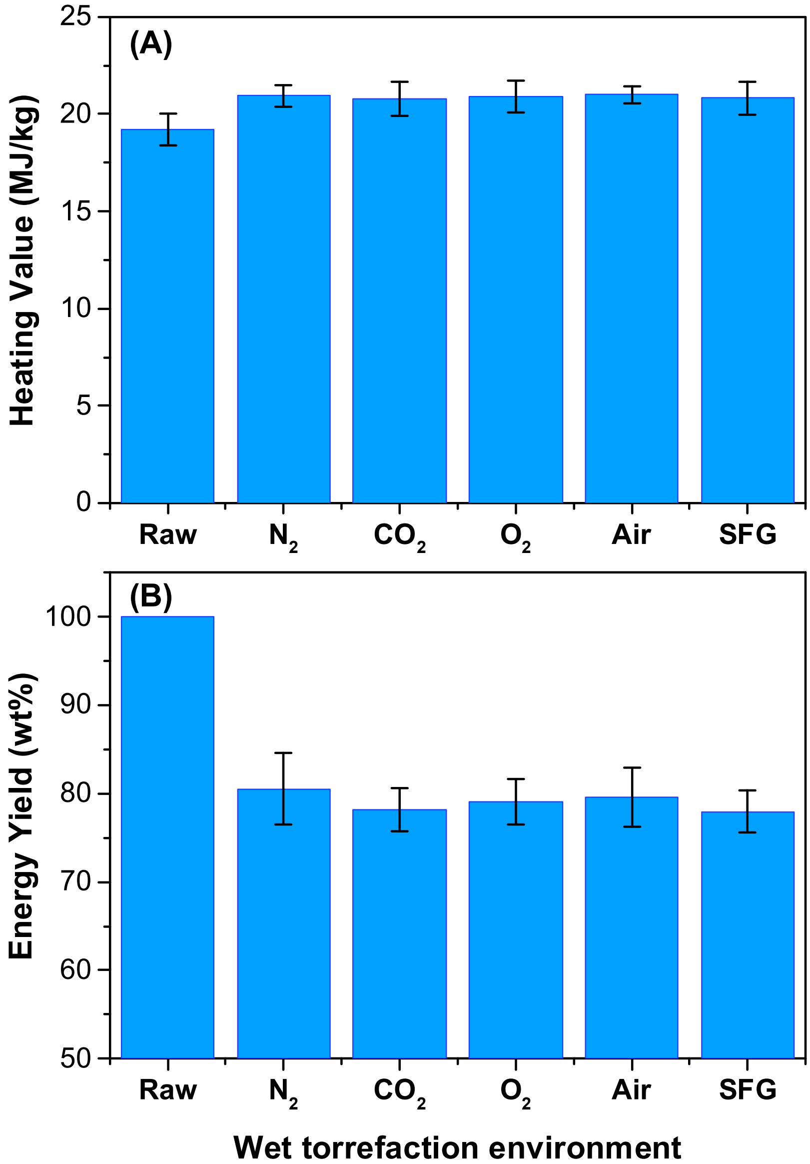

Figure 5. *Heating value (A) and energy yield (B) of raw Korean pine and its hydrochars obtained from WT in different atmospheres*

4 Discussion

In the DT process, the presence of oxidative gases such as CO_2 and O_2 has negative effects on the solid yields and its properties, for example, lowering the biochar yield and reducing the biochar fuel properties [19, 20, 22, 23, 25, 26]. However, these are different from WT, in which the atmosphere has insignificant effects on the yields and most of the hydrochar fuel properties, except for the ash contents, even though the oxidative gases are present in high concentrations up to 100%. These interesting observations open the possibility of utilizing waste heat from hot flue gas or hot air for WT without any major effects on the product yields and fuel properties.

The cause for the insignificant effects of WT may be due to the presence of water, which might become a "shield" to prevent biomass from direct contact with non-inert gases. In the case of DT, non-inert gases directly contact the biomass, and unwanted oxidative reactions occur, which reduces both the product yield and fuel properties. In another study [17], reductions in the yield of hydrochars produced by WT at 70 bar in CO_2 compared to that in N_2 were observed. However, the effect of CO_2 on the hydrochar properties is marginal. It is also worth noting that the saturated pressure of water at 200 °C is 15.5 bar and the selected pressure in this study (~20 bar) is much lower than that in the previous work (70 bar). The lower pressure helps reduce the amount of CO_2 penetrating the water medium, according to Henry's law of solubility of gas in liquid. Consequently, it decreases the acidic catalytic effect and has insignificant influences on the yields of the hydrochars obtained from WT in different gases and gas mixtures.

An interesting point of WT in the presence of CO_2 is the significant reduction in the hydrochar ash contents, compared with that in other atmospheres without CO_2. This is due to the acidic catalyst effect, which can efficiently remove inorganic ash elements and convert them into water-soluble compounds. This could be an attractive option to produce clean hydrochar with regard to ash elements.

5 Conclusions

WT of Korean pine (*Pinus koraiensis*) in various gases and gas mixtures, including N_2, CO_2, O_2, air, and SFG, has been conducted to examine the effects of these atmospheres on the yields and fuel properties of the produced hydrochars. The results show several enhancements in the fuel properties of the hydrochars including decreased O/C ratio and improved heating value. An interesting finding from this study is that the presence of oxidative gases such as O_2 and CO_2 in WT at low pressure has an insignificant effect on the yields and fuel properties of the hydrochars. The outcomes from this study could encourage the utilization of waste heat sources for WT at industrial scale to reduce the operating cost.

Appendix

Appendix A1. *Results from one-way ANOVA for different variances*

Characteristic	p-value	F-value	F critical	RMSSE
Hydrochar yield	0.7710	0.4492	3.4780	0.3870
Proximate analysis				
Ash content	0.0023	9.1047	3.4780	1.7421
Volatile matter content	0.9551	0.1575	3.4780	0.2291
Fixed carbon content	0.6679	0.6052	3.4780	0.4491
Ultimate analysis				
Carbon content	0.9997	0.0109	3.4780	0.0602
Hydrogen content	0.9350	0.1959	3.4780	0.2555
Nitrogen content	0.9417	0.1835	3.4780	0.2473
Oxygen content	0.9991	0.0198	3.4780	0.0813
Heating value	0.9965	0.0394	3.4780	0.1145
Energy yield	0.8246	0.3704	3.4780	0.3514

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