HYDROTHERMAL PROCESSING OF CHINESE TALLOW TREE (*TRIADICA SEBIFERA* SYN. *SAPIUM SEBIFERUM)* BIOMASS[1]

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ABSTRACT

Woody biomass holds great potential as a renewable source for bio-based materials, feed stocks, and energy. One particular herbaceous species that merits further investigation is the Chinese tallow tree (*Triadica sebifera* [syn. *Sapium sebiferum*]), which has extremely rapid growth, distribution, and has become noxious in the United States. This work explored the potential of Chinese tallow tree (wood/bark, leaves, and seeds) as a raw material for bio-based chemical and energy production using hydrothermal (HT) conversion. Seeds were HT-treated in the whole and ground states. Ground wood/bark, leaves, and seeds yielded similar aromatic compound assemblages after HT treatment. Ground seeds yielded unique minor by-products and did not contain naphthalene, which was present in the other tissue types. Whole HT-treated seeds yielded a material that resembled asphalt in appearance, odor, and chemical properties but did not produce any phenol. In contrast, ground seeds did not yield any particulate matter and had substantial amounts of phenol. In terms of elemental analysis of the ground and whole seed HT samples, the residues had increased C:H weight ratios. With regards to the energy input/output of this work, the HT treatment had a fairly neutral effect on energy content of the tallow seeds. The energy values of the tallow seeds are much higher than those typically reported for hardwood stemwood.

Keywords: Bark, biomass, Chinese tallow tree, leaves, solid wastes, supercritical water, transformation/recovery/recycling, wood.

INTRODUCTION

Reductive hydrothermal transformation of biomass

Biomass, including degraded and partially transformed wastes, has residual energy that can be recovered and used for human needs. Biomass transformation into chemical feedstock, product, or mixture requires that some energy be invested into the system to 1) render the physical characteristics of the material amenable to the operating parameters of the treatment, and 2) to perform chemical reactions that provide desired end products (Catallo and Shupe 2003; Catallo et al. 2004). In the many approaches to biomass conversion, these can include mechanical processing, heat, catalysts, and/or special chemicals

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(solvents, reactants), which can be expensive, toxic, or otherwise hazardous.

Hydrothermal (HT) refers to near- and super-critical water systems (e.g., 350°–450°C, 200–500 bar) under anoxic and reducing conditions. HT conditions are achievable in many settings, including field deployable units, modest in terms of energy required, and require abundant and recyclable materials/reactants (i.e., water and in-organic salts). HT treatments of all types of bio-mass (i.e., protein, cellulose, chitin, starch, DNA, bacteria cells, yeast cells, diatom cells, decommissioned preservative-treated wood wastes, grass (*Spartina alterniflora*), pond weeds (*Lemna* sp.), and municipal sewage slurry) have resulted in transformation of the bio-molecules to mixtures of gas and liquid phase aromatic and aliphatic chemicals (Catallo and Junk 2001; Catallo and Shupe 2003; Catallo et al. 2004).

Previous research on HT treatment of decom-missioned waste wood containing residual pre-servatives showed that (1) wood biomass can be transformed into useful hydrocarbon mixtures, and (2) HT conditions can be achieved that al-low for recovery or destruction of residual pre-servative chemicals (Catallo and Shupe 2003; Catallo et al. 2004).

Chinese tallow tree

Woody crops hold great potential for biomass production (Hansen 1992). Short rotation woody crops are mainly used as a fiber source for the pulp and paper industry. Fast-growing woody crops are also beneficial because they lessen the harvest demand on public and private forests. This is particularly important in the U.S. South because federal regulation and forest land own-ership have caused a shift in fiber production from the Pacific Northwest to the South. The pri-mary factor contributing to this shift is concern over endangered species and other possible ad-verse environmental impacts. (USDA FS 1997).

There is a huge global demand for wood fiber. The U.S. domestic consumption of wood fiber is equivalent to the consumption of a 6-ft. (1.8-m) long 2- × 4-in. (5- × 10-cm) piece of lumber per person per day. Globally, this consumption is equivalent to 2-ft. (0.6-m) of a 2- × 4-in. (5- × 10-m) piece of lumber per person per day (McLain 2001).

Coincident with the increased demand for wood products, a major change has occurred in the species composition of the forests of the southeastern U.S. In particular, Chinese tallow tree (*Triadica sebifera* [syn. *Sapium sebiferum*]) is widespread and noxious (USDA NRCS 2004). The species is extremely well adapted to numer-ous environments (Conner 1994; Donovan et al. 1988; Jones and McLeod 1989; 1990), and there are no known diseases that debilitate it (Scheld et al. 1980). Also, it produces aboveground bio-mass at a significantly faster rate than most other tree species (Harcombe et al. 1993; Rockwood et al. 1993) and is able to establish a dense stand quickly.

Chinese tallow tree was introduced to North America in the hope that the oil-rich (e.g., 4,700 L/ha) seeds would become a cash crop as in Asia. This seed oil production far exceeds other oil seed crops including cottonseed, soybean, rape, sunflower, safflower, and peanut, which produce 150–800 L/ha (Kane et al. 1987; Hsu et al. 1994; Xu et al. 1991; Liu et al. 1998). In 1986, the USDA Office of Critical Material des-ignated the Chinese tallow tree as one of four-teen promising new crop species for American farmers to grow new hydrocarbon stocks and industrial materials (Kane et al. 1987; Hsu et al. 1994; Liu et al. 1998).

Forest inventory surveys in the 1990s re-vealed 1,035,000 forest land acres with at least 1.6-ft.^{2} (0.15-m^2) basal area/ac (≥1.0-in. (2.5-cm) dbh (diameter at breast height)/ac) or dbh/0.40 ha in Chinese tallow for three regions: east Texas, Louisiana, and Mississippi. These Chi-nese tallow "occupied" forests contained 32,655,929 tonnes or 5.74 tonnes/ha—an amount equivalent to 7.4% of the tree biomass in these forests. Examination by forest type and other incidental observations of seedlings (<1.0 in. (2.5 cm) dbh) suggest a potential for substan-tive growth in sparsely forested regions and dis-persal in or along primary drainages (Rudis et al. 2005).

As a result of its inherent chemical properties and its proliferation in the landscape, tallow tree is an excellent candidate for biomass conversion using HT treatment. The objective of this research was to examine the potential of Chinese tallow trees for HT chemical conversion. The goals included (1) product chemical analysis and comparison of HT-treated ground leaves, bark, and whole seeds; (2) identification of structural and ultrastructural features of HT-treated versus untreated whole seeds (effect of substrate surface area size on products); and (3) an evaluation of the energy value btu/lb (J/kg) of untreated seeds as compared to the seeds following HT treatment.

MATERIALS AND METHODS

Three representative Chinese tallow trees measuring 6–8 m in height and 15–20 cm dbh were harvested in October 2003 from a forested area in East Baton Rouge Parish, Louisiana. Immediately upon felling, the tree boles were separated into leaves, bark, wood, and seeds. The bark was removed using a ring debarker. All tree components except for the seeds were reduced to mulch with an industrial waste shredder. Each tree part, including the seeds, was further reduced to particles in a Wiley Mill that passed a 1-mm mesh screen. All components were then stored in air-sealed bags in a freezer at −12°C. The fruit is three-lobed with one seed in each lobe. Seeds are covered with a white waxy coating (University of Florida IFAS 2004). Seeds were used as collected and had the following mean characteristics: 9.4-mm length and 0.24-g mass.

HT treatment of ground and whole samples

Weighed particle samples (2.5 g) were added to 65-mL internal volume high pressure autoclaves in the presence of water above pH 10. Whole seeds experiments used between 10–20 seeds (*ca*. 2.5–5.0 g). The HT system was sparged with Ar for 1 h, sealed, and incubated at 400 ± 10°C/380 bar for 6 h including heat-up and cool-down. Once the samples reached the desired temperature (*ca*. 50 min), the heating element was turned off and the temperature maintained adiabatically.

Subsamples were weighed into cellulose thimbles (Whatman, Springfield Mill, UK;10 × 50 mm) and micro-Soxhlet extracted with dichloromethane (DCM) to determine the semivolatile hydrocarbon (HC) content of the untreated wood. The resulting extract was dried over anhydrous sodium sulfate and analyzed by gas chromatography–mass spectrometry (GC-MS).

GC-MS: Shimadzu QP500 GC-MS; DB-5 capillary column (30 m; 0.25 mm id; 0.25 μm film); injector 250°C; temperature program 50°C (4 min) ramp 4°C/min to 250°C (10 min); sampling rate 2 Hz and mass acquisition range 50–300 amu in the full scan mode. Analyte transfer to the mass spectrometer source was at 280°C and the source energy 70 eV. Target ions spanned the inclusive molecular weight range between toluene (92 amu) and coronene (300 amu). Product identification was performed using a) comparison of experimental data with authentic standards, b) interpretation of mass spectra (molecular ions, isotopic structures, and logical fragment losses), and c) comparison of spectra with computerized libraries of mass spectra. Mass spectra were considered acceptable if there was a signal:noise ratio of >3 for the base peak of interest, and minimal background interference with respect to isotopic clusters and fragments. MS tuning (PFTBA–perfluorotributylamine, a standard calibrant) was performed at least once daily, and all MS analyses for comparison were conducted under the same tune. Digital background subtraction and chromatographic overlay algorithms were used for further evaluation of GC-MS data from standards and extracts.

Energy determination

Energy content was determined in btu and converted to joules (J) using the following conversion: 1 btu = 1,055.0559 J. Energy content was determined using ASTM D5865 (formerly ASTM D2015) for gross calorific value of coal

and coke (ASTM 2004). Preweighed, oven-dry whole seed samples (approximately 1 g) were placed in a bomb calorimeter, which then was pressurized with oxygen. Both untreated controls and post HT-treated samples were analyzed. The tarry, asphalt-like residue following HT treatment was air-dried prior to testing. The bomb was placed in a 20°C water bath and the sample ignited via a firing wire. The energy released was estimated by the change in vessel air temperature. The detection limit was 50 btu/lb (116,320 J/kg) on a 1-g sample. To account for possible volatile loss, which was found to be minimal, we determined the amount of moisture present on a separate aliquot of the sample.

Scanning electron microscopy

Starting materials and post-HT treatment samples were placed on 1-cm (id) Al stubs covered with C-adhesive tabs (EMS, Washington, PA) and then coated with Au/Pd using a Hummer V DC sputter-coater. Liquid hydrocarbon samples (from the fine particle treatments) were allowed to evaporate until the consistency was tarry, followed by mounting on the C-adhesive strips and coating as above. The samples were visualized using an FEI Quanta 200 Electron Microscope (Peabody, MA) at magnification 80; 600; 1,200; 5,000; and 20,000.

RESULTS AND DISCUSSION

HT treatment

HT treatment of ground leaves, seeds, and wood/bark and whole seeds resulted in the production of gas phase and liquid (semi-volatile) hydrocarbon (HC) mixtures (Fig. 1a–c). All ground materials were completely transformed to HCs (there were trace amounts of particulate residue), but whole seeds had significant amounts (>25% wt.) of solid residue resembling asphalt in appearance, odor, and physical properties. Extracts of control ground leaves, seeds, and wood/bark showed no detectable HCs in GC-MS analyses (data not shown). The major HT products included alkyl benzenes, phenols,

indanes; and these were comparable between leaves, wood/bark, and seeds. The compound distributions in the ground seed and wood/bark were very similar in terms of all resolved HCs and peak elution patterns (e.g., peak groups 8, 9, 14, and 17 in Fig. 1 b and c). The HT-treated ground leaf residues were significantly different than the ground seed and wood/bark: the n- and branched chain HCs were better developed in leaf samples, while the seeds and wood/bark contained more abundant and diverse assemblages of 2–4 ring polycyclic hydrocarbons (PCHs), which were not detected in the leaf treatments (e.g., naphthalene was found in substantial amounts in all whole and ground HT treatments except for the leaves). This finding was unexpected and the reasons are not clear.

Data in Fig. 2a show the GC-MS analyses of products in extracts from HT treatment of whole tallow seeds. The relative abundances of several major extractable HCs (alkyl benzenes- and branched HC) yield were increased relative to the ground seeds, e.g., there were no PCHs heavier than C_2–naphthalene (e.g., 1,2-dimethylnaphthalene) detected, whereas in the ground seeds, PCHs heavier than pyrene were detected. Substrate size had a strong influence on HT product distributions (Fig. 2 b and c). As shown in the figure, phenol is prominent in ground seed HT extracts, but is relatively depleted in whole seed HT treatments run identically.

In sum, the most obvious differences in HT products between ground and whole seed treatments were: (1) the ground seed mass was entirely transformed into gas phase and liquid HC mixtures, while substantial amounts of the whole seed mass (albeit transformed) remained after HT treatment as an asphalt-like material (Fig. 3); (2) there was a size effect on the physical and chemical composition of residues, e.g. extractable hydrocarbon residues, and tarry solids; and 3) treatment of ground leaves yielded a mixture that was comparable to ground wood/bark and seeds in terms of several light aromatic compounds but otherwise significantly different in terms of relative abundances of n- and branched HCs and PCH. Substrate size influences the kinetics of the HT reactions. Smaller
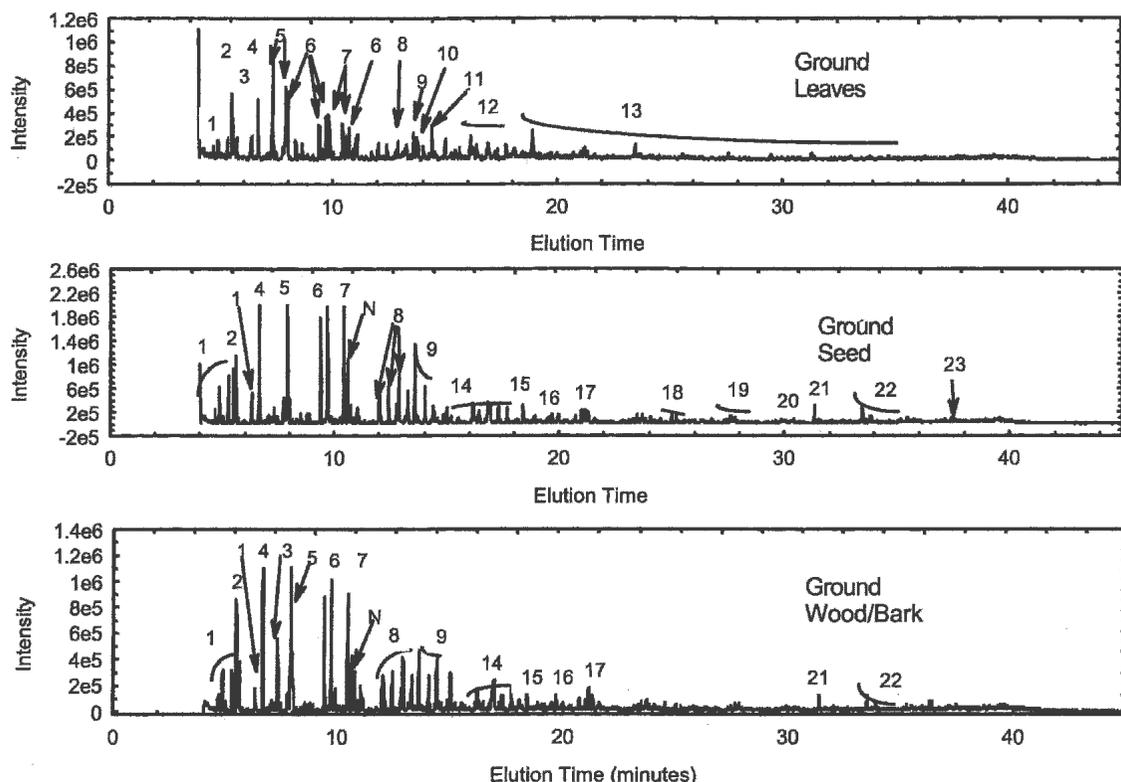
FIG. 1. Total ion chromatogram (TIC) of extracts from HT-treated (a) ground leaves, (b) ground seed, and (c) ground wood/bark. Compounds: 1. C_3-Benzenes. 2. Phenol. 3. n-hydrocarbon. 4. Indane. 5. C_1-Phenols. 6. C_1-Indanes. 7. C_2-Phenols. N = naphthalene. 8. C_2-Indanes. 9. C_1-Naphthalene. 10. Indole. 11. C_1-Benzaldehyde. 12. Hydroxyindanes (6 isomers). 13. n- and branched hydrocarbons. 14. C_2-Naphthalenes. 15. Acenaphthene. 16. C_3-Naphthalenes. 17. C_1-Fluorene. 18. Anthracene/Phenanthrene. 19. C_1- Anthracenes/Phenanthrenes. 20. Fluoranthene. 21. Pyrene. 22. mass 216, e.g., C_1-Pyrenes and benzofluorenes. 23. mass 228, e.g., Triphenylene, Benzanthracene, Chrysene.

substrates clearly have a higher surface area and faster reaction times.

Figure 3 shows SEM prints of control and HT-treated whole seeds and the liquid residue of HT-treated ground seed. Comparison of Fig. 3A and B with Fig. 3C and D indicates substantially increased HT treatment (1) substantially (>10 fold) decreased the size of the seed, (2) increased the apparent porosity (surface area) of the residual structure, and 3) eliminated virtually all of the initial (pretreatment) surface structure. The surface of the HT-treated seeds closely resembled a pyrogenic carbon soot matrix with "grapes in a cluster" morphology comprised of spherical subunits on the order of 3–5 microns in diameter (Catallo et al. 2001). Figures 3E and F are from HT treatment of ground seed and

show a characteristic DCM-soluble oily residue with no particulate residue observed. The HT treatment of whole seed yielded extractable hydrocarbon mixtures and a residual tarry material, which is a carbonized, soot matrix containing absorbed and adsorbed organic chemicals of which approximately 40% by mass was readily soluble in DCM.

Elemental analyses

Elemental analysis of the ground seed (pretreatment) vs. the ground and whole seed residues after HT treatment is shown in Table 1. Following both ground and whole seed HT treatments, the residues had much increased C:H weight ratios, with control C:H ratios of 7.3
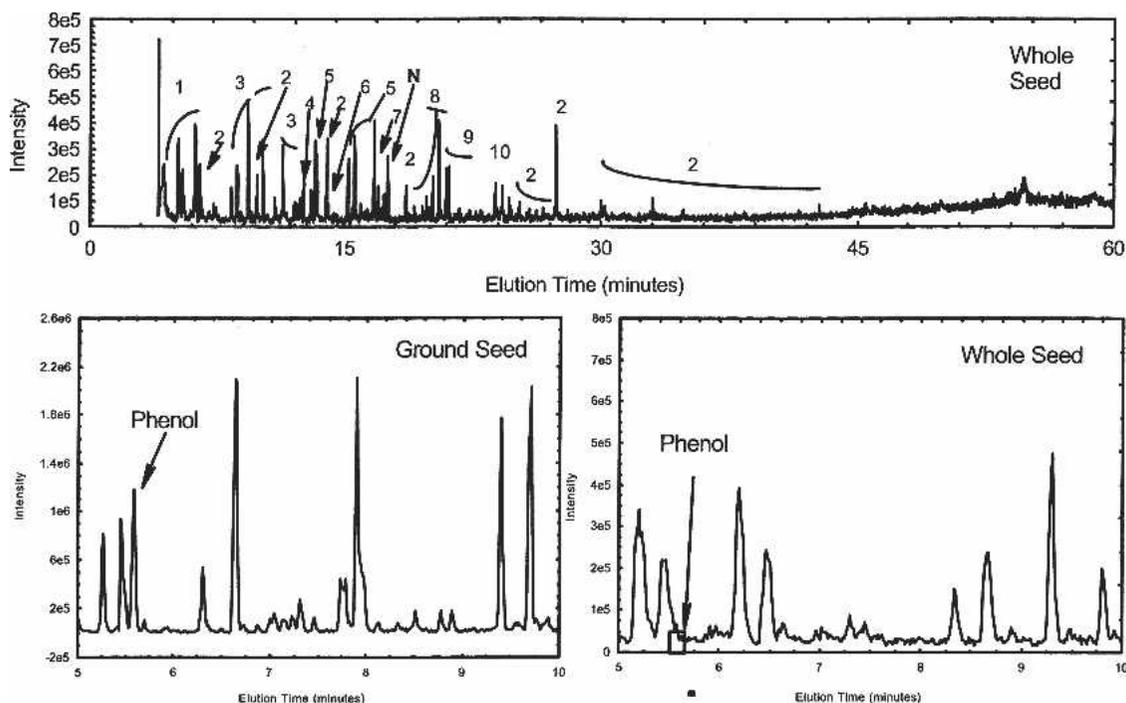
FIG. 2. Total ion chromatogram (TIC) of extracts from HT-treated (a) whole seeds; (b) and (c) showing exploded view of TIC of extracts from HT-treated ground seed and whole with respect to phenol. Compounds: 1. C_2-Benzenes. 2. n- and branched hydrocarbons. 3. C_3-Benzenes. 4. Indane. 5. C_1-Indanes. 6. C_1-Phenols. 7. C_2-Indanes. N. Naphthalene. 8. C_5-Benzenes. 9. C_1-Naphthalenes. 10. C_2-Naphthalenes.

(control) and 10.1 in the products. This trend is consistent with chemical analyses showing distributions of aromatic hydrocarbons in the products (e.g., the C:H mass ratio of starch is approximately 7.0, while that of benzene is 12. Interestingly, the levels of nitrogen in whole seeds doubled after HT treatment (values increased by a factor of 10–20). This showed that the tarry solids remaining after the treatment of the whole seed had residual plant nitrogen in transformed bio-molecules of high molecular weight (e.g., lignin).

In the case of whole seeds, it appears that the nitrogen (occurring in bio-molecules such as lignin) is not reacted completely or at all in the bulk of the whole seed, which at least partially explains the high concentration of nitrogen. In the ground seeds, where there is no particulate residue after HT treatment, we believe that the nitrogen is lost as inorganic nitrogen species such as nitrate, nitrogen gas, and nitric acid.

Energy analyses

A quantitative comparison of the heat of combustion values of untreated tallow seeds versus post HT-treated seeds is found in Table 2. The HT treatment had virtually no effect on the energy content of the residue from the treated seeds. Thus, while completely transformed, the chemical residues from HT treatment still had high levels of chemical potential energy. It is noteworthy in this regard that the energy of the tallow seeds is much higher than that typically reported for hardwood stemwood. The average heat of combustion of 23 hardwood species was found to be 7,827 btu per oven-dry pound (Karschesy and Koch 1979). The tallow seeds yielded much higher energy values than wood values reported by Karschesy and Koch (1979) and as such, have potential as a bio-based raw material/feedstock for advanced materials, particularly energy. The high energy values for the
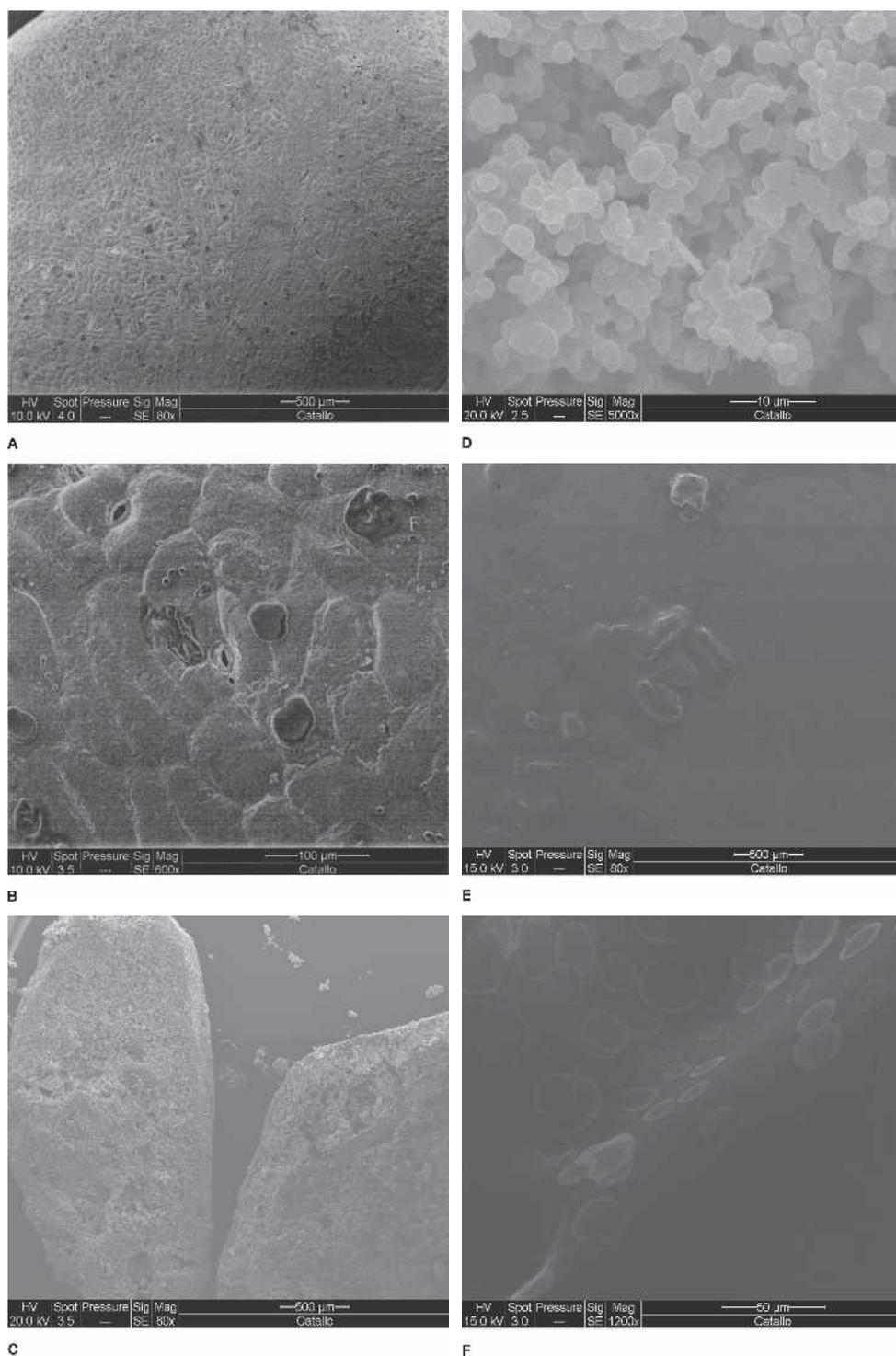
FIG. 3.   Scanning electron micrographs of tallow material showing (a) control whole seed (80×), (b) control whole seed (600×), (c) HT-treated whole seed (80×), (d) HT treated whole seed (5,000×), (e) HT-treated ground seed (80×), (f) HT treated ground seed (1,200×).

TABLE 1. *Elemental analysis of the control (not HT treated) and experimental (HT treated) Chinese tallow tree biomass.*[1]

Element	Control seeds	Control wood/bark	HT-treated seeds ground (oil)	HT-treated seeds whole (air dried)	HT-treated seeds whole (tarry solids)
Carbon wt. %	56.76[2] (1.15)[3]	46.21 (0.02)	77.26 (0.39)	83.86 (1.02)	70.71 (0.52)
Hydrogen wt. %	7.33 (0.17)	5.92 (1.13)	12.32 (0.58)	4.66 (0.19)	6.01 (0.40)
Nitrogen wt. %	0.83 (0.14)	0.08 (0.01)	0.19 (0.10)	2.50 (0.02)	1.98 (0.16)

[1] The known standard (atropine) was run in duplicate with error of less than 0.06%.
[2] Means based on n = 2.
[3] Numbers in parentheses denote standard deviations.

TABLE 2. *Comparison of the heat of combustion values of untreated tallow seeds versus post HT-treated seeds.*

	btu/lb	kJ/kg
	Untreated	
Mean	11,560	26,894
Standard deviation	5.73	
	HT treated	
Mean	11,581	26,942
Standard deviation	5.80	

tallow seeds can likely be attributed to the inherently high concentration of fats and oils in the seed. Eberhardt and Shupe (2004) found the mean alcohol-toluene extractive content of the seeds to be 43.1%.

CONCLUSIONS

Previous work by the authors has shown that in general wood is a suitable substrate for HT transformation into usable hydrocarbon assemblages. The objective of this research was to examine the effect of substrate size and tissue type on the products of HT-treated Chinese tallow tree. The data included (1) product chemical analysis and comparison of HT-treated ground leaves, bark, and whole seeds, (2) structural and ultrastructural features of HT-treated versus untreated whole seeds (effect of substrate surface area size on products), and (3) an evaluation of the energy value btu/lb (J/kg) of untreated seeds as compared to the seeds following HT treatment.

The major chemical products of the HT treatment were similar between all ground tissue types, particularly with respect to substituted benzenes, phenolic compounds, and indanes. There were differences in the minor products; for example, HT-treated leaf samples failed to show naphthalene, which was present in the other ground tissues.

The size of the substrate material affected the physical and chemical properties of the products. For example, whole seed HT treatments yielded a tarry asphaltic material and gas and liquid phase hydrocarbon mixtures which were depleted in phenol relative to the ground samples, which were transformed completely into gas and liquid hydrocarbons. Scanning electron micrographs indicated that the HT treatment greatly decreased the size of the seed, increased the apparent porosity (surface area) of the residual structure, and eliminated virtually all of the initial (pretreatment) surface structure.

With respect to the energy input/output of this work, the HT treatment had a fairly neutral effect on energy content of the tallow seeds, providing a set of products that are chemically transformed and still possess high potential energy. The energy values of the tallow seeds are much higher than those typically reported for hardwood stemwood.

The results of this and related work suggest that bio-based energy and bio-based chemicals have great potential as a renewable source of raw material to satisfy many industrial uses. The principal negative aspect of this technology is economic feasibility, which currently is unknown except for preliminary data, which are encouraging. The HT process has potential as an environmentally friendly fossil fuel alternative. Moreover, some potential end-uses of the HT products from tallow tree could include refinement of liquid hydrocarbons (e.g., petrochemicals), direct collection and reuse of gas phase hydrocarbons (e.g., for heat generation), and

varied uses of asphaltics (e.g., road tars and chemicals). In the presence of "green" (e.g., solar, hydroelectric, and geothermal) and other (e.g., industrial waste heat, heat exchangers) sources of energy, the biological feeds for the HT process can be farmed or culled from bio-wastes and treated in a completely "green" and nearly completely recycled system. As a result, contemporary, rather than fossil carbon, is employed for industrial purposes and there is no net CO_2 loading of the atmosphere.

Keys for future research and commercialization will be reaching the desired temperature as quickly and economically as possible, and maximizing the utility of the adiabatic energy in the system after reaching the desired temperature in the reactor.

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