

FINAL REPORT

Use of Improved Densification Conditions for Producing High Fuel Content Products from Biomass Processed by Torrefaction, Hydrothermal Carbonization, and Various Densification Methodologies

by:

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TABLE OF CONTENTS

LIST OF TABLES.....	iv
LIST OF FIGURES.....	v
SYNOPSIS.....	1
SECTION I	2
Introduction – Scoping Studies.....	2
Process and Equipment Limitations Identified.....	2
Outside Storage Limitations.....	3
Compaction Protocol Limitations	4
Influence of Briquette Geometry.....	5
Lessons Learned from Hydraulic Ram Briquetting	6
Preliminary Binder Evaluations.....	7
Influence of Tar and Wax Components on Ash Fusion Properties and HGI Index	9
Influence of Pressure and Binder Dosage with Button Face vs Concave Die at Three- Second Retention Time.....	10
Strategies to achieve Moisture Resistance with Steam Explosion	11
Investigation of Coating Strategies for Imparting Moisture Resistance.....	13
Hydrothermal Carbonization (HTC) – Influence of Time, Temperature, and Grind Spec Across Birch, Pine, and Gum Tree from Australia.....	14
Binding Properties and Moisture Resistance of Steam-Stripped Torrefied Feedstock.....	16
Moisture Resistance of Briquettes with Increasing Inclusions of HTC Binder.....	18
Commercial Pellets Formulated with Commercial HTC Material.....	20
Hydro Mulch for Protection of Bulk Piles of Pellets or Briquettes	21
Scoping trial for use of our birch bark as binder to T-wood.....	22
Effective Densification Requires Manipulation of Many Variables	23
SECTION II	24
Investigation of Physical Properties of Densified Products.....	24
Pre-Treatment Protocol.....	24
Hot Briquetting	24
Cold Briquetting	26
Binders	26
Procedure.....	26
Drop Durability.....	26

Moisture Uptake	27
Particle Distribution	28
Kansas Durability.....	28
Crush Strength	29
Moisture Penetration Index (MPI)	30
Microwave Steam Production	30
Hydraulic Press.....	31
Results	32
Briquette Aging Results – 5 Day Cure vs 30 Day Cure	35
Overall Property Evaluation for the Various Blends Tested	38
Discussion	38
Particle Distribution	41
Bulk Density	41
Conclusions	41
SECTION III	43
Use of Briquette Blends in Stationary Hearths of Two One-Quarter-Scale Steam Locomotives at the Milwaukee Zoo.....	43
Conclusions	51
Future Work.....	51
Acknowledgement	52
SECTION IV	53
Overcoming the Densification Challenges of Torrefied and Hydrothermally Carbonized Biomass: Key Learnings and Challenges	53
Executive Summary.....	53
Evolution of Development at NRRI	54
Lessons Learned.....	55
Process and Equipment Limitations Identified	55
Outside Storage Limitations.....	56
Compaction Protocol Limitations	57
Influence of Briquette Geometry.....	58
Hydraulic Ram Briquetting.....	59
Preliminary Binder Evaluations.....	60
Influence of Tar and Wax Components on Ash Fusion Properties and Hardgrove Grindability Index (HGI)	62

Influence of Pressure and Binder Dosage with Button Face vs Concave Die at 3 Second Retention Time.	63
Strategies to achieve Moisture Resistance with Steam Explosion	64
Coating Strategies for Imparting Moisture Resistance.....	65
Hydrothermal Carbonization – Influence of Time, Temperature, and Grind Specification across Birch, Pine, and Gum Tree from Australia	67
Moldability and Release Character of HTC.....	68
Binding Properties and Moisture Resistance of Steam Stripped Torrefied Feedstock	68
Moisture Resistance with Increasing Inclusions of HTC Binder.....	70
Commercial Pellets Formulated with Commercial HTC Material.....	72
Hydromulch for Outdoor Storage of Bulk Piles of Pellets or Briquettes	72
Scoping Trial for Use of Outer Birch Bark as Binder to Torrefied Wood	73
SYLVA Corporation Torrefied Kiln Runs, February 2011.....	74
Co-firing Trial at a major 56,000 Tons/Year Pulverized Coal Utility	76
Advanced Biofuels can be produced in Various Agglomerated Shapes	78

LIST OF TABLES

Table I-1: Binder comparisons for durability and moisture resistance.	8
Table I-2: Comparison of the influence of binder on grindability, chemistry, and ash fusion point.	9
Table I-3: Durability performance of Oriox 255/PVA binder with concave versus button die.	10
Table I-4: Hydrothermal carbonization processing conditions for various species.	14
Table II-1: Formulations tested.*	32
Table II-2: Summary Table of NRRI Formulation Performance to Consumer Charcoal and Industry Standard Coal (Five-Day Cure).....	40
Table III-1: Analytical comparisons for the fuel products tested.	48
Table IV-1: Operability parameters used in the commissioning trial runs on B-100 briquetter.....	60
Table IV-2: Preliminary Scoping Trials Characterizing Various Binders	61
Table IV-3: Characterizations of tar and wax binder variations and the control.	62
Table IV-4: HGI ratings.	62
Table IV-5: Durability performance of Oriox 255/PVA Binder with concave versus button die.	63
Table IV-6: Various HTC characterizations.....	67
Table IV-7: Typical calorific values for the torrefied wood operating with a kiln breach temperature of 321°C (610°F) over a sustained operating period of 14 hours at 63.6 kg/h (140 lb/h).*	75

LIST OF FIGURES

Figure I-1: Influence of degree of torrefaction on grindability of torrefied particulate matter.	3
Figure I-2: Comparison of agglomerate integrity when in contact with wet coal.....	3
Figure I-3: Carver press used in initial densification and the influence of ‘time at pressure’ on agglomerate shape.	4
Figure I-4: Comparison of die geometry in tests with carver press.....	5
Figure I-5: Comparison of geometries for die shape.	6
Figure I-6: B-100 Hydraulic ram compactor.....	6
Figure I-7: Parameters used in trials with the B-100 ram briquetting machine.....	7
Figure I-8: Concave (a) and button die (b) produced compacts.	10
Figure I-9: Moisture resistance of steam-exploded blends.	11
Figure I-10: Moisture resistance of steam-exploded blends.....	12
Figure I-11: Pictorial of various alternative means for adding moisture resistance.	13
Figure I-12: Basic scheme for converting biomass using hydrothermal carbonization in a 5.5-liter Parr reactor.	15
Figure I-13: Illustration of the moldability HTC processed biomass.....	15
Figure I-14: Comparison of moisture uptake of various samples including steam-stripped materials.	16
Figure I-15: Microscopic and SEM analysis of steam-stripped torrefied wood versus commercially produced material.....	17
Figure I-16: Comparison of moisture resistance of briquettes with increasing HTC levels.	18
Figure I-17: Pictograms of moisture resistance of steam-stripped blends.	19
Figure I-18: Pictograms of pellets made at commercial scale from blends of HTC and torrefied wood.....	20
Figure I-19: Use of hydro mulch as a moisture barrier for fuel piles.....	21
Figure I-20: Inclusion of HTC processed outer birch bark in torrefied blends and moisture resistance.....	22
Figure I-21: Key variables for forming acceptable briquettes.	23
Figure II-1: Basic production circuit used to produce torrefied fuels at NRRI’s Renewable Energy Laboratory.....	25
Figure II-2: Basic densification circuit used to produce torrefied fuels at NRRI’s Renewable Energy Laboratory.....	25
Figure II-3: Drop durability testing.....	27
Figure II-4: Moisture uptake illustration.....	27
Figure II-5: Determining particulate size distribution.....	28
Figure II-6: Kansas durability illustration.	29

Figure II-7: Crushing strength test configuration.....	29
Figure II-8: Moisture penetration illustration.....	30
Figure II-9: Microwave heating of blend to correct moisture level.....	31
Figure II-10: Briquetting of prepared mixture.	32
Figure II-11: Average crush strength in pounds force at fracture.	33
Figure II-12: Tumbling durability survival of 500 grams of each sample using the Kansas State Tumbling Can.	33
Figure II-13: Average drop durability after 5 drops and 20 drops.....	34
Figure II-14: Greatest percentage of moisture uptake over a 24-hour immersion period.	34
Figure II-15: Average drop durability for samples at 5 days versus 30 days measured after 5 drops and 20 drops.	35
Figure II-16: Comparison of Kansas Tumbling Durability for briquettes aged 5 days and 30 days.	36
Figure II-17: Comparison of Crush Strength in pounds force for briquettes aged 5 days and 30 days.	36
Figure II-18: Greatest percentage of moisture uptake over a 24-hour immersion period.	37
Figure II-19: Particle distribution of oak versus pine after torrefaction.....	38
Figure III-1: Steam engines used during the stationary hearth test programs at the Milwaukee Zoo.....	44
Figure III-2: Steam engines heating up boilers using typical coal fuels.....	44
Figure III-3: Briquettes supplied for first tests at the Milwaukee Zoo.....	45
Figure III-4: Briquettes used during the second test at the Milwaukee Zoo.	45
Figure III-5: Comparison of fuel briquettes and coal used to power steam locomotives.	46
Figure III-6: Sparklers emitted during full drafting of the boiler.	47
Figure III-7: Flame characteristics from ram briquettes.	47
Figure III-8: Hearth coverage under low-draft conditions.....	49
Figure III-9: Combustion of fuel briquettes under full draft conditions.	49
Figure III-10: Comparison of flame characteristics for coal and torrefied biomass fuels.	50
Figure III-11: Comparison of stack emissions in using coal or coal/torrefied fuel blend.	50
Figure III-12: Stack emissions for 100% torrefied biomass briquettes (steam release also shown at right of stack.....	51
Figure IV-1: Evolution of torrefaction development over time.....	54
Figure IV-2: Evolution of hydrothermal carbonization research.....	55
Figure IV-3: Torrefaction levels and the effect on grindability.....	56
Figure IV-4: White wood pellets versus torrefied wood pellets stored on wet coal bed.....	56
Figure IV-5: Carver press.....	57
Figure IV-6: Limitations of “time-at-pressure” for Carver press scale-up.	57

Figure IV-7: Concave die (a) and button die (b) effect on briquette density gradient.	58
Figure IV-8: B-100 ram briquetter.	59
Figure IV-9: Sparklers from stack and finished briquettes.	59
Figure IV-10: Concave briquette versus button briquette.	63
Figure IV-11: Steam explosion binders B or D, and torrefied wood formulations and moisture resistance characterization.	65
Figure IV-12: Coating and densification strategies to achieve moisture resistance.	66
Figure IV-13: Raw material to energy mud – the hydrothermal carbonization process.	67
Figure IV-14: HTC waffle briquettes.	68
Figure IV-15: Moisture uptake of steam-stripped briquettes versus HTC briquettes.	69
Figure IV-16: Microscopic and SEM analysis of steam-stripped torrefied wood versus commercially.	70
Figure IV-17: 24-Hour immersion of commercial HTC versus birch bark waste HTC briquettes formulated with commercially torrefied wood.	71
Figure IV-18: 24-Hour immersion of commercial HTC versus birch bark waste HTC briquettes formulated with steam-stripped torrefied wood.	71
Figure IV-19: 24-hour immersion of high BTU/lb pellets.	72
Figure IV-20: State of pellets and briquettes after wetting hydromulch covering.	73
Figure IV-21: 24-hour immersion of outer birch bark binder and torrefied wood briquettes at various inclusions.	74
Figure IV-22: Photographic detail of the modified Sylva Rotary Kiln showing tor gas flair to atmosphere and material flow collection into steel drum.	76
Figure IV-23: System used at coal utility.	76
Figure IV-24: Sulfur reduction through use of biomass.	77
Figure IV-25: Mercury versus Time. Reduction of mercury utilizing biomass.	77
Figure IV-26: Various products can be made from the advanced biomass fuel products.	79

SYNOPSIS

The Natural Resources Research Institute is engaged in work to develop demonstration-level production of solid biofuel densified products that can be stored outside, have high bulk densities for ease of logistical transport, have good handling characteristics that minimize dust generation, possess grindability that is like coal used in power plants, and have fuel contents that match or exceed sub-bituminous coal levels. During the work, two pretreatment technologies have been investigated for concentrating the energy content of raw biomass. These include: torrefaction using an indirectly fired rotary kiln process at the demonstration level and hydrothermal carbonization at the bench and pilot scale. The Institute has also collaborated with Syngas technologies on a pilot-scale moving bed, directly heated steam-based process at the pilot scale and next year will install this technology at the demonstration scale. A key factor in showing the full technical feasibility of using the pretreated materials is to demonstrate that the produced particulate fuel products can be densified to a level that allows good logistical and handling practices to be routinely attained. It has been found that hydrothermally carbonized processed materials can be agglomerated using a variety of densification devices including pelleting and briquetting in a repeatable and practical manner using commercial densification equipment with and without the use of binders.

However, torrefied materials have proven to be much more difficult to densify using a variety of densification equipment, especially as the degree of torrefaction increases. Uniformly torrefied materials at high energy level appear to be especially difficult to densify but have the attributes of high fuel value and good grindability, with very little residual fiber content compared to less-torrefied material or steam-exploded biomass.

Therefore, the work undertaken and explained in the following discussion has been conducted and shows that highly torrefied materials can be satisfactorily densified to produce high-energy-content products that have good physical properties, possess acceptable moisture resistance, low ash, sulfur and mercury content, and have bulk densities that can lead to improved logistics. The densification practices involve optimizing overall process conditions on an integrated systems basis and include moisture level, densification pressure, mix preparation pressure, and the use of appropriate binders when required. The densification system that seems to show the greatest promise for the highly torrefied materials is briquetting. Work will continue in examining other densification options and in improving the conditions used and discussed in this report.

SECTION I

Introduction – Scoping Studies

In the course of execution of Task 4 of the CAWES program, various scoping studies were undertaken to determine various potential methods to produce converted biomass products that simultaneously possessed high fuel value, low levels of tramp impurities and ash, possess satisfactory physical properties to resist degradation in handling and transportation, have excellent grindability, and have the ability to resist moisture related material breakdown. In prior work at the Natural Resources Research Institute (NRRI) it had been found that torrefied biomass is often difficult to densify when the degree of torrefaction is enhanced to allow higher fuel value products to be attained. As noted in Task 2 results from the actual use of torrefied pellets in various commercial scale tests, under-torrefied materials also can lead to grinding difficulties in existing bowl or roller mill pulverizers, and this is thought to be caused by the incomplete breakdown of the fibrous nature of the woody biomass. In order to investigate these areas, a series of scoping trials were conducted using both bench- and demonstration-scale processing at NRRI. The following discussions summarize what was learned in conducting these studies.

Process and Equipment Limitations Identified

One of the equipment limitations our team identified early on was the grindability of the material. If the degree of torrefaction of the material was too low or not uniformly torrefied, the tenacity of the under-torrefied fibers could not be properly ground with existing equipment. This was especially true for typical bowl mills used in commercial power plants. We found that the use of a turbulizer equipped with the appropriate impacting surfaces was effective in grinding the torrefied materials into desired particulate distributions that aided downstream compaction. In addition, the turbulizer could accomplish both mixing and grinding in a single unit operation simultaneously.

Error! Reference source not found. demonstrates the variability when grinding different levels of torrefied materials with a turbulizer. Under-torrefied material is too tenacious and results in grinding difficulty, as evidenced by the lighter particles pointed out and the resulting smaller portion of fines generated across the turbulizer. Although the turbulizer is not really designed to be a grinder, our team found that with an acceptable torrefaction level, the size distribution of material exiting the turbulizer was very suitable for forming durable and moisture-resistant briquettes.



Figure I-1: Influence of degree of torrefaction on grindability of torrefied particulate matter.

Outside Storage Limitations

When co-firing pellets with wet coal, white wood pellets have limitations when compared to torrefied wood pellets or briquettes. White wood pellets degrade and disintegrate when they come in contact with wet coal particles. Conversely, torrefied pellets or briquettes, if they are stored in contact with wet coal particles, survive and do not disintegrate readily. These attributes are clearly displayed below (Figure I-2), where white wood pellets, after being placed onto a simulated bed of wet coal particles, wick up the moisture, expand, and disintegrate back to their original state. Torrefied pellets survive and can be seamlessly handled in conventional coal handling systems.

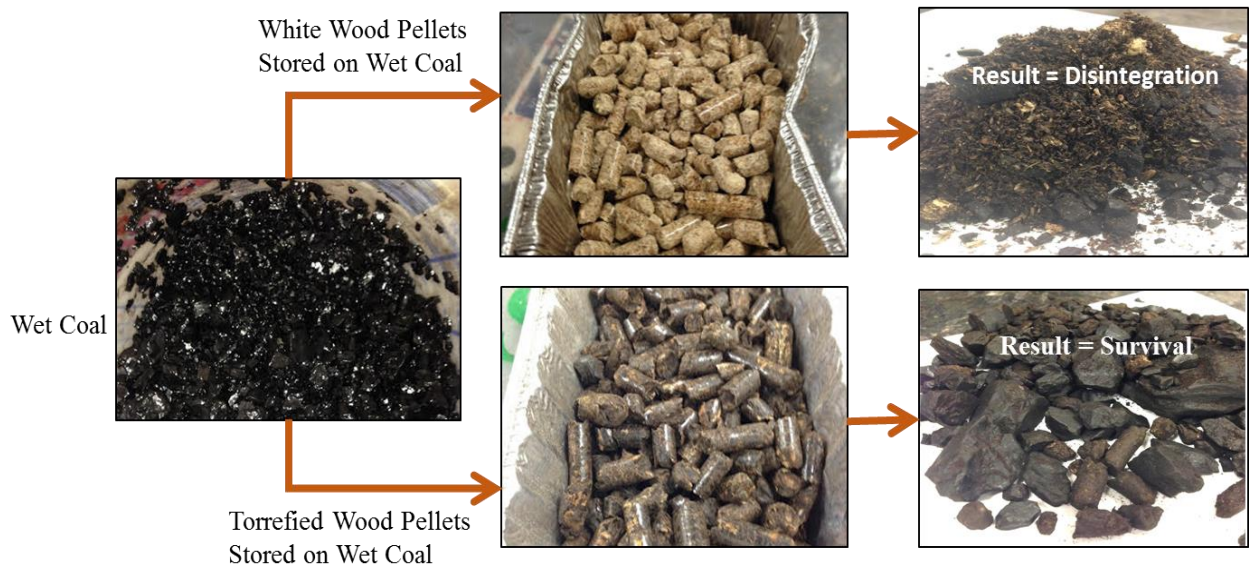


Figure I-2: Comparison of agglomerate integrity when in contact with wet coal.

Compaction Protocol Limitations

Compaction protocols must be developed for each specific material across several variables including mixing time, temperature, moisture, particle size, and retention time at specified pressure. One of the limitations our team identified when using an axial type Carver press was the ‘time-at-pressure’ parameter. In general, for most materials, a 15-second retention ‘time-at-pressure’ is required to get durable moisture-resistant briquettes. This makes scale-up to commercial equipment challenging, where ‘time-at-pressure’ parameters are of the order of 1–2 seconds. Generally, the Carver press is useful to determine the minimum pressure to generate a durable moisture-resistant briquette at targeted particle size, moisture content, and binder type taken at a minimum 15-second retention time at pressure in the die. Then, as commercial runs are initiated, fine tuning may be required where more binder or more heating time prior to compaction is required.

The limitation of ‘time-at-pressure’ for briquettes formed at 2, 5, 15, 30, and 60 seconds is shown graphically in Figure I-3. As shown, the briquettes really do not begin to hold their shape until after 15 seconds.

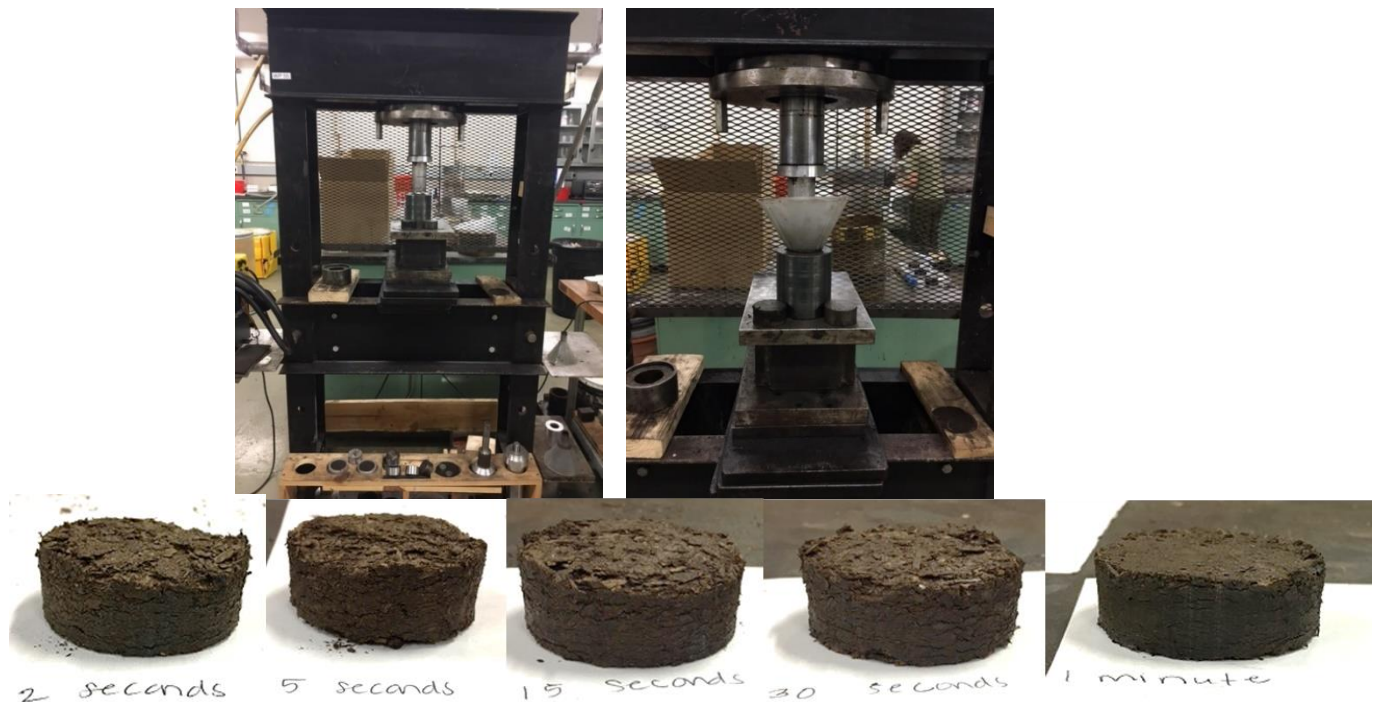


Figure I-3: Carver press used in initial densification and the influence of ‘time at pressure’ on agglomerate shape.

For most torrefied materials, the ideal conditions for forming a durable moisture-resistant briquette is a pre-heat temperature 88°C – 104°C (190°F – 220°F), a grind specification of 70% passing 30 mesh (0.595 mm), and a retention time of about 15–30 seconds. The ideal moisture content is approximately 4–12% when loading the feedstock into the die cylinder. However, there can be significant differences noted across different material types. For example, our team has found that with red oak, a 4% moisture content, 70% passing 30 mesh (0.595 mm), and a temperature of 88°C (190°F) makes durable moisture-resistant briquettes on a

commercial briquetter (Komarek B220B). However, to achieve the same degree of moisture resistance with red pine, a much higher temperature in the 99°C – 104°C (210°F – 220°F) range is required.

Influence of Briquette Geometry

One of the limitations our team identified for briquette geometry when using the Carver press was that, with a concave die, a density gradient is evident across the depth of the briquette. Since compaction was happening in a single downward motion, a natural gradient formed where material with greater density was at the bottom portion of the die compared to the top portion. This characteristic would surface when testing durability, where the top portion of the briquette would abrade more easily than the lower portion. To help ameliorate this effect, raised buttons were affixed to the top and bottom portion of the concave dies. This helped to eliminate the gradient across the die and helped to improve durability significantly. This not only eliminated areas of lower density (on the cylindrical surfaces) but also created air space within a pile of briquettes, thereby enhancing uniform combustibility (Figure I-4 and Figure I-5).

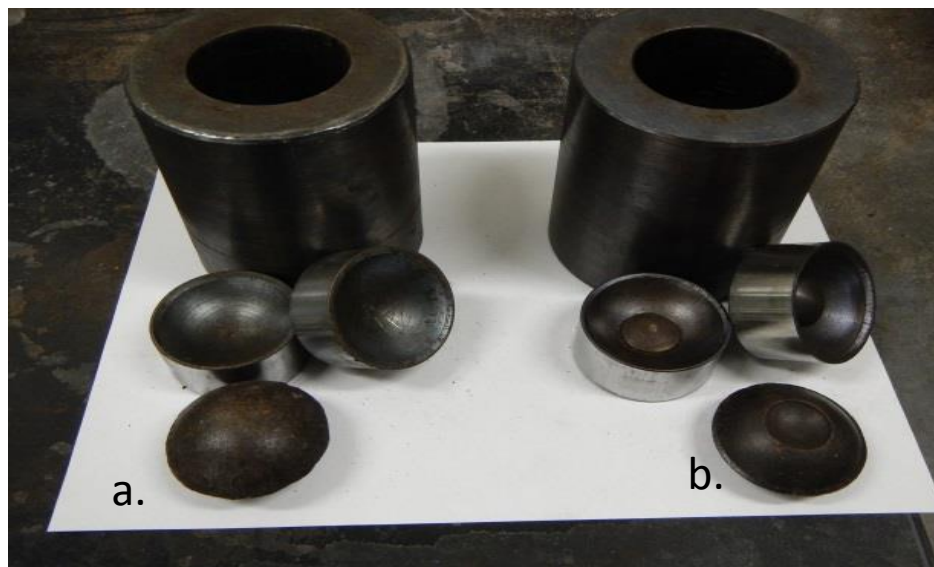


Figure I-4: Comparison of die geometry in tests with carver press.



Figure I-5: Comparison of geometries for die shape.

Lessons Learned from Hydraulic Ram Briquetting

The B-100 hydraulic ram compactor (Figure I-6) was used to determine densification parameters of a variety of torrefied biomass feedstock with the use of a poly vinyl alcohol (PVA) binder system (Figure I-7). Steam-exploded biomass as well as high- to moderately torrefied feedstocks were used. The operability parameters were determined, as well as the limitations of the B-100. Parameters identified included: hydraulic pressure, system operating clamp pressure, and feed rate needed to form viable briquettes. Grind specification was also evaluated; torrefied feedstock was hammer milled to a -9.525 mm (-3/8 inch) size specification. The take-away learnings from these initial compaction trials was that the -9.525 mm (-3/8") grind specification combined with axial compaction was inadequate when actual briquettes were burned in a quarter scale steam locomotive engine. These cylindrical fuel briquettes were found to expand significantly when burned in an updraft, fixed-bed boiler. This resulted in significant “sparklers” emitting from the stack of the steam locomotive. The sparklers were deemed a safety hazard in the operation. As a result, the team abandoned this compaction scenario when end use is in updraft, fixed-bed boiler systems. The sparklers were due to the lack of through compaction densities obtained with this method. As the outer surface of the compact was topochemically combusted, the interior particulate matter was not held together and could be pulled apart by the bed draft. More on this trial will be discussed in Section III of this report.



Figure I-6: B-100 Hydraulic ram compactor.

Operability parameters used in the commissioning trial runs on the B-100 briquetter.

	Operating Clamp	Clamp setting		System
	Pressure	low high	Feed rate setting	Operating
Run	psig	psig	(Unitless)	Pressure, psig
1	800	1700/2300		0.4 2000-2300
2	800	1700/2300		0.4 2000-2300
3	1000	1700/2300		0.4 2000-2300
4	1000	1700/2300		0.4 2000-2300
5	1000	1700/2300		0.4 2000-2300
6	1000	1700/2300		0.4 2000-2300
7	500	1700/2300		0.4 2000-2300

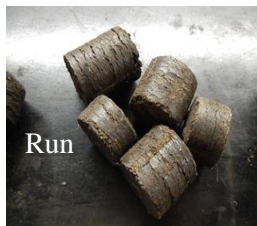


Figure I-7: Parameters used in trials with the B-100 ram briquetting machine.

Preliminary Binder Evaluations

Preliminary scoping trials were conducted across a variety of binder types with durability and moisture-resistance (MPI) factors as metrics. These scoping trials were undertaken to identify those binder systems that yielded both durability and moisture resistance. A moisture penetration index was used as a screening tool also to eliminate those binders that would imbibe a droplet of water in less than 3,600 seconds. Those binder types that yielded MPIs of > 3,600 seconds were selected as candidates for future investigation and scale-up. These initial scoping trials indicated that guar gum, wax, hydrothermally carbonized (HTC) Birch Bark, HTC Wood, HTC Switchgrass, and steam exploded wood could impart moisture resistance if included in a formulary and were prepared properly (see Table I-1). The typical binder content employed was 10%.

Table I-1: Binder comparisons for durability and moisture resistance.

Binder	Durability Factor	Moisture Resistance factor	Notes
None	Excellent when formed at high temperature and grind spec. of < 20 mesh (0.841 mm)	Very good at 177°C (350°F) forming temperature and grind spec of < 20 mesh (0.841 mm)	Hot forming at 177°C (350°F) with normal energy content achieves moisture resistance, not so good with high energy content material ³ MPI > 3600 s
Calcium lignosulfonate	Good	Poor	MPI < 120 s
Sodium lignosulfonate	Good	Poor	MPI < 120 s
CaO	Good	Poor	MPI < 120 s
Ca(OH) ₂	Good ³	Poor	MPI < 120 s
CaCO ₃	Good	Poor	MPI < 120 s
Molasses	Good	Poor	MPI < 120 s
CaO/molasses	Good	Poor	MPI < 120 s
Xantham gum	Good ¹	Poor	Good binder for high btu material
Guar gum	Good ²	Poor	Good binder for high btu material
Bentonite	Good	Poor	MPI < 120 s
Asphalt tar	Good	Excellent	MPI > 18,000 s
Paraffin wax	Good	Excellent	MPI > 18,000 s
Hydrothermally modified birch bark tar	Good	Excellent	MPI > 18,000 s
Hydrothermally modified wood	Good	Excellent	MPI > 18,000 s
Hydrothermally modified switch grass	Good	Excellent	MPI > 18,000 s
Steam exploded wood	Good	Excellent	MPI > 18,000 s
Cellulose acetate	Good	Poor	MPI < 120 s
Brewex	Good	Poor	MPI < 120 s
Sodium silicate	Good	Poor	MPI < 120 s
Recycled tear-off shingles (TOS)	Good	Poor	Could be better if silica is removed

^{1,2}Good at low dosage; ³high energy content material still has poor MPI even with high temperature.

Influence of Tar and Wax Components on Ash Fusion Properties and HGI Index

One of the concerns our team had with some of the binder systems was their potential negative influence on ash fusion properties, HGI, and sulfur content. A series of experiments were conducted at 10% binder inclusion with torrefied wood and the sulfur, heating value (HHV), and ash fusion temps measured (Table I-2).

Table I-2: Comparison of the influence of binder on grindability, chemistry, and ash fusion point.

Binder ¹	HGI	HHV (btu/lb)	Sulfur (%)	Ash (%)	Ash Fusion (oxd)	Ash Fusion (red)
Control	30	9,176	0.06	1.89	>2640°F	>2640°F
Crafco Tar (fossil)	27	9,515	0.261	3.46	>2640°F	>2640°F
B.B.Tar (natural)	38	9,615	0.12	1.62	>2640°F	>2640°F
5828 Tar (fossil)	19	9,785	0.475	2.67	>2640°F	>2640°F
Paraffin Wax (fossil)	13	10,174	0.042	2.34	>2640°F	>2640°F

¹All binders tested at 10% dosage formed under similar conditions
1Btu = 1.055 kJ; 2640°F = 1,449°C 10,000 BTU/lb = 23,210 kJ/kg

HGI Reading	Difficulty Level
< 40	Very Hard
40–60	Hard
60–80	Moderately Hard
80–100	Soft
100–120	Very Soft
< 120	Extremely Soft

All the binders generally showed an improvement in heating value, and surprisingly, none of them lowered the ash fusion temperature compared to a control. There was no notable trend evident in the HGI data other than to note the particles were found to stick to the grinding balls, and this likely influenced the HGI test results. It was concluded that the HGI was not an appropriate test for evaluating binders due to the limitations of binder particles adhering to the grinding balls, and this factor merits more consideration when choosing test materials. Paraffin wax, for example, may stick onto the grinding balls, resulting in difficulty grinding and material recovery. This would lead to creating erroneous measurements. Craftco Tar and P5828 Tar are asphalt tars, resulting in higher sulfur content than the others listed. The increased ash content of Crafco Tar, P5828 Tar, and Paraffin wax all had higher percentage of ash content, likely because they were formed from a fossil fuel base material. Our conclusions are that HGI should only be used for well-torrefied and uniformly torrefied materials without any binder. The influence of binder sticking to the ball charge combined with under-torrefied or non-uniformly torrefied material only makes the HGI test yield erroneous harder to grind values. The validity of any HGI tests should be highly scrutinized whenever it is used on torrefied materials.

Influence of Pressure and Binder Dosage with Button Face vs Concave Die at Three-Second Retention Time.

Table I-3: Durability performance of Oriox 255/PVA binder with concave versus button die.

2% Oriox 255/PVA Binder with Concave 2-inch die	
Compaction Pressure (Bar)	Durability, % Survival
240	53.3
479	96.8
958	98.4
1438	99.4
2% Oriox 255/PVA Binder with Button 2-inch die	
Compaction Pressure (Bar)	Durability, % Survival
240	63.7
479	98.8
958	99.4
1438	98.0
0.7 % Oriox 255/PVA Binder with Button 2-inch die	
Compaction Pressure (Bar)	Durability, % Survival
958	98.1
958	99.5

Note: All samples used three-second die retention time, 958 bars, microwave heat to 10% moisture to 88°C – 99°C (190°F – 210°F).

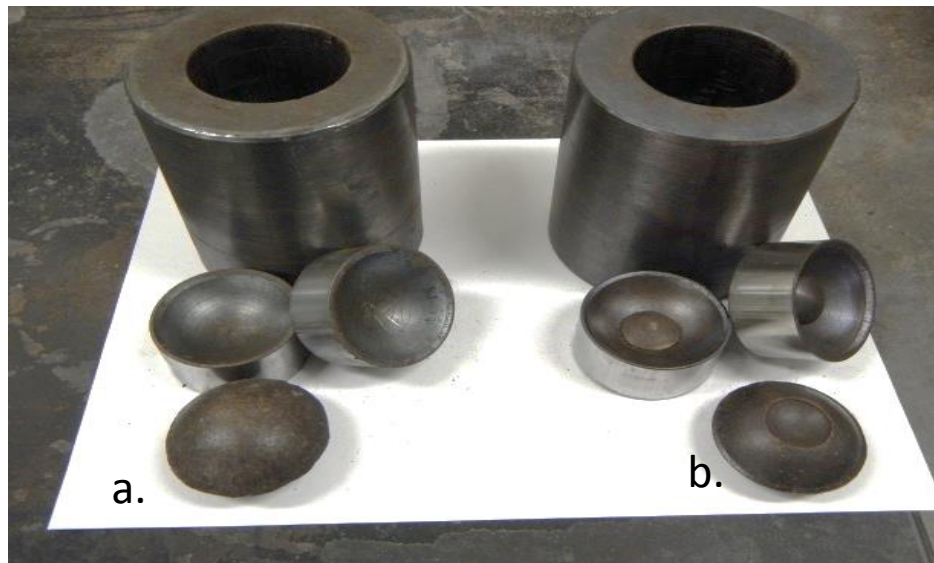


Figure I-8: Concave (a) and button die (b) produced compacts.

The Oriox 255/PVA binder system was evaluated at 2% and 0.7% dosage levels with a button face and concave die (Figure I-8). Our findings revealed better durability was achieved with the button-faced die, and binder dosage could be reduced to the 0.7% level. In addition,

the incorporation of the PVA allowed retention times to be reduced to the three-second range. All the above tests were done at three-second die retention in comparison to the earlier standard of 15 seconds.

Strategies to achieve Moisture Resistance with Steam Explosion

The objective for these experiments was to determine the use of a steam-explosion feedstock as a binder to torrefied wood to impart moisture resistance. Our team received steam-exploded loblolly pine from Herty's steam explosion lab. The steam explosion conditions for the two samples received were: 165°C, 30 min, and severity factor = 3.4(B) and 175°C, 90 min, severity factor = 4.2(D). Briquettes consisted of 100% binder and were 50.8 mm (2 inch) diameter (concave die), made individually on a 50-ton H-Frame press at 1,000 bar and under microwave preheating conditions of 88°C – 99°C (190°F – 210°F). The briquettes were then placed on a simulated wet coal bed to observe any moisture uptake or disintegration. This experiment demonstrated that steam explosion at 165°C – 175°C was not enough to impart moisture resistance, as virtually all the briquettes wicked up moisture from the simulated coal bed, expanded significantly, and opened any bonds that were formed during the compaction process. This was also confirmed during the commissioning of the hydraulic ram B-100 where steam-exploded feedstock was used as a binder to torrefied wood briquettes. These briquettes, when exposed to moisture, would also disintegrate and break apart (see Figure I-9 and Figure I-10).



100% SE binder B before and after wet coal exposure. Moderate disintegration noted after 3.81% moisture uptake.



30% SE binder D/70% T-Wood before and after wet coal exposure. Severe disintegration noted after 6.86% moisture uptake.

Figure I-9: Moisture resistance of steam-exploded blends.



30% SE binder B/70% T-Wood before and after wet coal exposure. Severe disintegration noted after 9.81% moisture uptake.



20% SE binder D/80% T-Wood before and after wet coal exposure. Severe disintegration noted after 9.34% moisture uptake.



10% SE binder B/90% T-Wood before and after wet coal exposure. Severe disintegration noted after 6.93% moisture uptake.

Figure I-10: Moisture resistance of steam-exploded blends.

Investigation of Coating Strategies for Imparting Moisture Resistance

Various samples of binders, briquette formulations, and coatings were tested to determine if any were feasible to impart moisture resistance. The description and results are noted in the picture captions (Figure I-11).



A thin coating of hot wax can be spray coated onto the briquette surface, but the coating is easily sloughed off upon extensive handling.



Hot forming the briquettes at 200°C (392°F) imparts significant moisture resistance, but this temperature range is outside the

capabilities of commercial compaction equipment.



Application of a powder wax followed by heating can be used to imbibe wax into the surfaces of the briquette, but upon weathering the coating may slough off.



Double compaction agglomeration: anticipated lower surface area with the addition of HTC binder and moisture resistant individual particles.

Instead, pre-compacted particles resulted in uneven mating surfaces—providing a conduit for moisture penetration between particles—and the resultant product did not have good moisture resistance.



Coarse ground particles versus fine ground particles offered no difference in moisture resistance when white wood pellets were torrefied, crumbled, and then re-agglomerated.



When white wood pellets are torrefied and then crumbled and sized, they offer excellent moisture resistance and low dust content. These crumbled and sized agglomerates could easily be stored outside.

But bulk density is relatively low at 32 lb/ft³.

Figure I-11: Pictorial of various alternative means for adding moisture resistance.

Hydrothermal Carbonization (HTC) – Influence of Time, Temperature, and Grind Spec Across Birch, Pine, and Gum Tree from Australia.

Table I-4: Hydrothermal carbonization processing conditions for various species.

Material	Processing Temperature (°C)	Processing Time (min)	Energy Content (BTU/lb)	Energy Content (kJ/kg)	Sulfur Content (%)	Ash Content (%)
Whole Tree Birch Chips	220	180	11,054	25,656	0.035	0.58
Birch Bark Waste — Unground	226	60	11,104	25,772	0.04	1.82
Birch Bark Waste — Ground	235	15	11,630	26,993	0.034	1.93
Birch Bark Waste — Unground	235	15	11,557	26,824	0.034	1.91
Pine Whole Tree Chips	235	15	11,153	28,886	0.015	0.54
Gum Tree — Whole Tree Chips	235	15	10,983	25,492	0.014	0.21

The influence of time, across a temperature range of 220°C – 235°C and across various material types and grinds, shows that the hydrothermal process results in energy contents exceeding 25,500 kJ/kg (11,000 BTU/lb) (Figure I-12). In addition, all the resulting materials are easily compactable with any densification device including ram compaction, rotary compaction, and ring and die pelletizing. This illustrates the robustness of the HTC conversion, regardless of wood type and whether it is chips or ground through a hammermill, the resulting material has significant coal-like properties and is easy to compact.



Figure I-12: Basic scheme for converting biomass using hydrothermal carbonization in a 5.5-liter Parr reactor.

One of the unique features of HTC materials is that it is very moldable and easily releases from complex die shapes (Figure I-13). The processed material can be readily consolidated into various shapes, with excellent physical properties.



Figure I-13: Illustration of the moldability HTC processed biomass.

Binding Properties and Moisture Resistance of Steam-Stripped Torrefied Feedstock

One of the important findings that our team continues to uncover is the surface preparation of the material through steam stripping. If super-heated steam is used as a sweep gas, the surface character of the material becomes more amenable to bonding.

The moisture-resistant properties of steam-stripped torrefied briquettes made from either black ash or tamarack were compared to 100% HTC briquettes (Figure I-14). Black ash had the most moisture resistance (lowest moisture uptake) among all the samples, including the HTC briquettes. The steam-stripped torrefied briquettes were formed using hot dies, whereas the HTC briquettes were formed using room temperature dies. This is a variable to consider when comparing the two sample classes. This experiment outlined the possibility that steam stripping when torrefying may prepare the feedstock to a level that may be comparable to HTC. The NRRI continues to investigate the use of steam stripping to have a cleaner resultant surface for the torrefied materials. It is also conjectured that the removal of condensable volatile matter using this technique reduces the flammability of the materials and increases natural particle to particle bonding during subsequent densification.

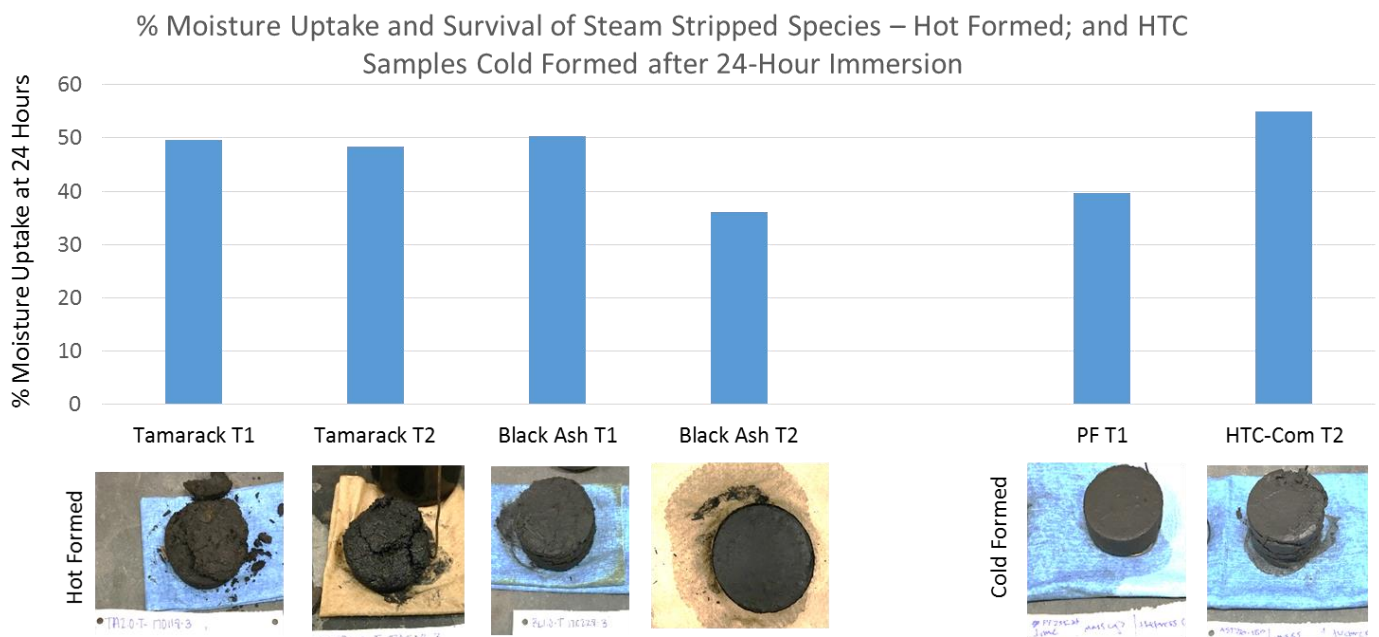


Figure I-14: Comparison of moisture uptake of various samples including steam-stripped materials.

As demonstrated in Figure I-15, the surfaces of steam-stripped torrefied wood and non-steam-stripped torrefied wood have variances. Pictures (a) and (b) depict the surface differences of densified material. Picture (a) has a much richer, brown color, whereas (b) has a darkened, black surface. This may indicate that steam stripping has removed some of the extractives and excess surface particles, essentially cleansing the surface to make it more easily densified. Photos (c) and (d) are SEM photos of steam-stripped and non-steam-stripped torrefied wood. From these images, it is shown that the cellular structures and cell walls of the steam-stripped sample (c) are significantly damaged during the torrefaction process, thus creating more surface area for binding. The surface in photo (d) shows more intact cell structures and fibrosity.

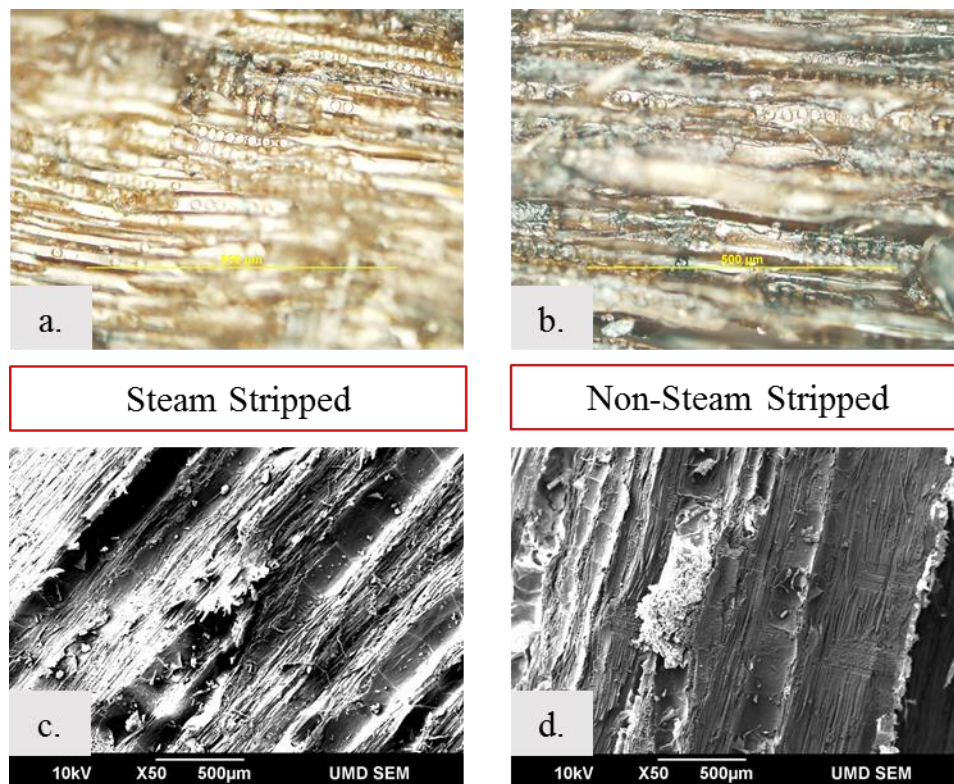


Figure I-15: Microscopic and SEM analysis of steam-stripped torrefied wood versus commercially produced material.

Moisture Resistance of Briquettes with Increasing Inclusions of HTC Binder

The two pictograms in Figure I-16 display the level of disintegration over a 24-hour period of briquettes composed of HTC (HTC-Com T2) and commercially torrefied wood at inclusion levels of 0–100%. The second set of briquettes was formulated from birch bark waste that was processed using HTC (PF T1) and commercially torrefied wood. The briquettes have increasing amounts of HTC added, going from 0% – 100% addition level. The results indicate that as higher percentages of HTC are added, more moisture resistance is attained. In addition, other results indicate that reactor retention times in HTC processing also influence moisture resistance. The first HTC (HTC-Com T2) was prepared at 220°C for 180 minutes, whereas birch bark waste HTC (PF T1) was prepared at 235°C for 20 minutes. Overall, the birch bark waste HTC briquettes imparted more moisture resistance at lower levels of inclusion than the other HTC briquettes. This is likely due to the difference in processing temperature. Higher processing temperature is a key variable in increasing the hydrophobicity of the produced HTC materials.

24 Hour Immersion of Torrefied Wood

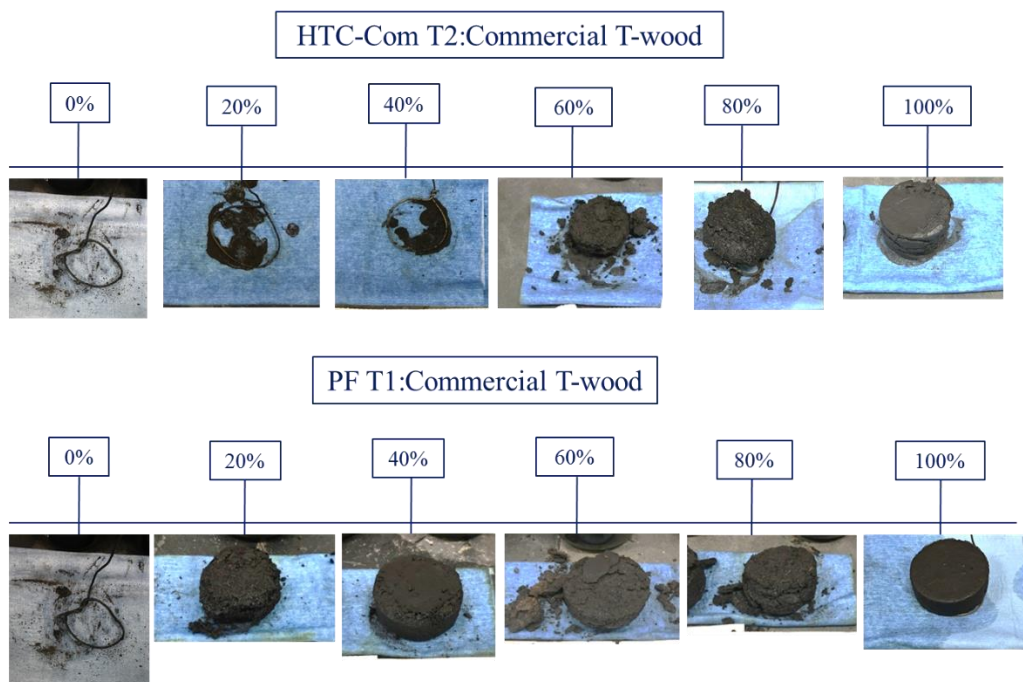


Figure I-16: Comparison of moisture resistance of briquettes with increasing HTC levels.

The two pictograms in Figure I-17 demonstrate the moisture resistance of briquettes composed of the two HTC species, as listed out in the previous paragraph, and combined with steam stripped torrefied wood. Between the two sample sets above, moisture resistance is seen with as little as 20% HTC inclusion; however, the briquettes formulated with the first HTC method had greater disintegration at 20% than those formulated with birch bark waste HTC. This points to the greater activity and functionality of the HTC formulated with the birch bark waste processed at higher temperature.

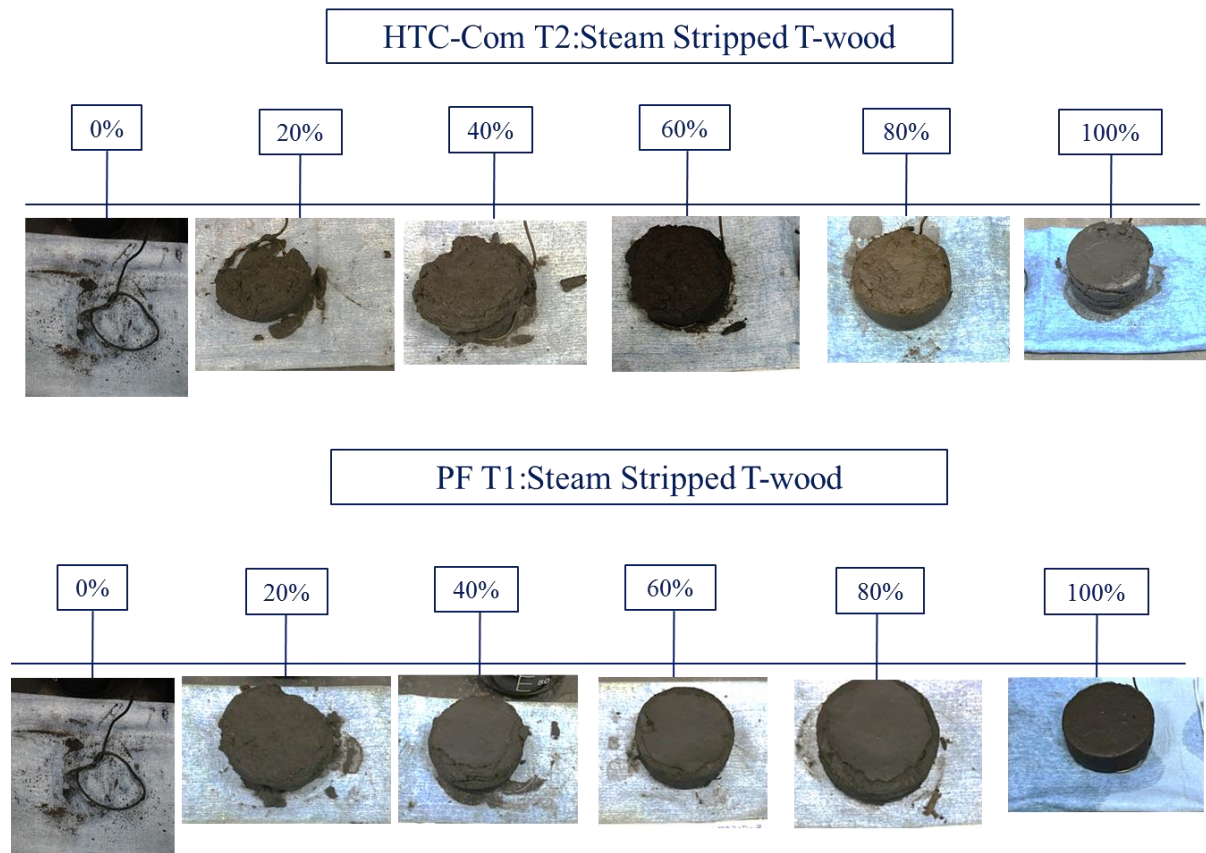


Figure I-17: Pictograms of moisture resistance of steam-stripped blends.

The key findings from the four samples sets highlight the importance of the HTC regime used in preparing the biomass. A higher temperature and lower retention time seem to impart more moisture resistance at lower inclusion levels than the HTC formulated at lower temperatures and a longer retention time. Also learned from these experiments is that steam stripping when torrefying versus traditional roasting can also impart moisture resistance. A clean surface preparation appears to be one of the drivers for achieving moisture resistance. Of the briquettes made using commercial HTC, those combined with steam-stripped torrefied feedstock had drastically more moisture resistance than those combined with commercially roasted torrefied wood.

Commercial Pellets Formulated with Commercial HTC Material

The NRRI contracted with a pilot HTC Facility to process approximately 500 kg of whole birch tree wood chips (bark-on) at 220°C for 180 minutes. This HTC material was then air dried and formed into pellet formulations at a commercial pellet facility. A commercial torrefied wood was used to blend with the HTC birch at ratios of 50% T-wood/50% HTC and 70% T-wood/30% HTC. All the pellets produced at the commercial facility survived direct submersion in water for 24 hours (Figure I-18). The specific die specifications that were used is proprietary.

State of High BTU (11,000 BTU/lb; 25.59 mJ/Kg) after 24-Hour Immersion in Water

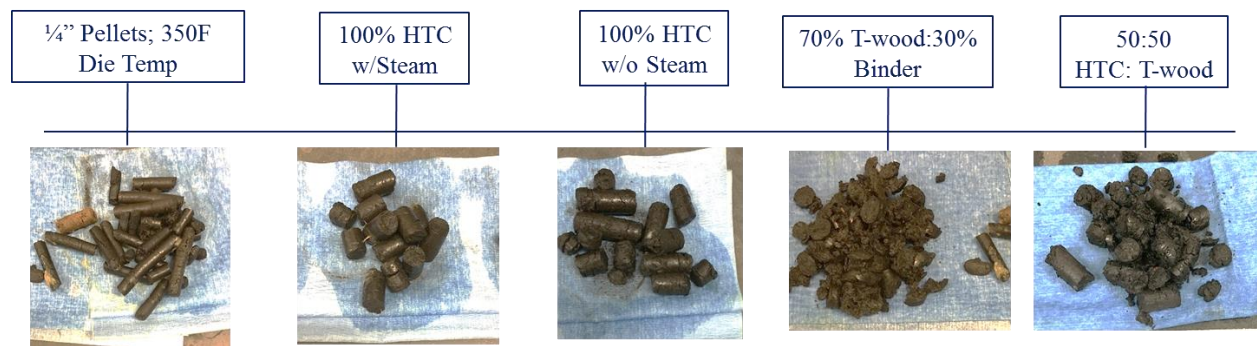


Figure I-18: Pictograms of pellets made at commercial scale from blends of HTC and torrefied wood.

Hydro Mulch for Protection of Bulk Piles of Pellets or Briquettes

In addition to the work already discussed that was done to increase the moisture resistance of the produced biomass fuel products, additional ideas were investigated to determine other ways to allow outside storage of materials with less moisture resistance. Scoping trials were investigated to explore the use of reactive hydro mulch as a protective barrier to cover bulk piles of pellets for outdoor storage. Initial hydro mulch blends incorporated 50% pulp mill residue with 50% refined wood fiber. A polysaccharide type binder system was used. Small plates of pellets were covered with reactive mulch formulation and allowed to harden. The hardened mulch layer was wetted repeatedly with water droplets to see how this protective layer would shed moisture and prevent moisture infiltration into the simulated pile. Inspection of the pellets in Figure I-19 indicated minimal disintegration. This type of a protection strategy could be implemented in more tropical or humid climates and as an extra measure to protect bulk piles from rain exposure and any subsequent material degradation. It also is likely a much more economical storage enhancement than requiring inside storage for the fuel products.



Figure I-19: Use of hydro mulch as a moisture barrier for fuel piles.

Scoping trial for use of our birch bark as binder to T-wood

One of the favorable aspects of outer birch bark is that it is highly concentrated in favorable organic compounds that impart hydrophobicity. Thus, birch bark, could be a good choice as a starting material for achieving moisture resistance. Our initial scoping trials revealed that even at inclusion levels of 10%, the HTC processed outer bark provided a high degree of moisture resistance. HTC outer birch bark material, in combination with conventional torrefied wood, all survived 72-hour immersion tests (Figure I-20).

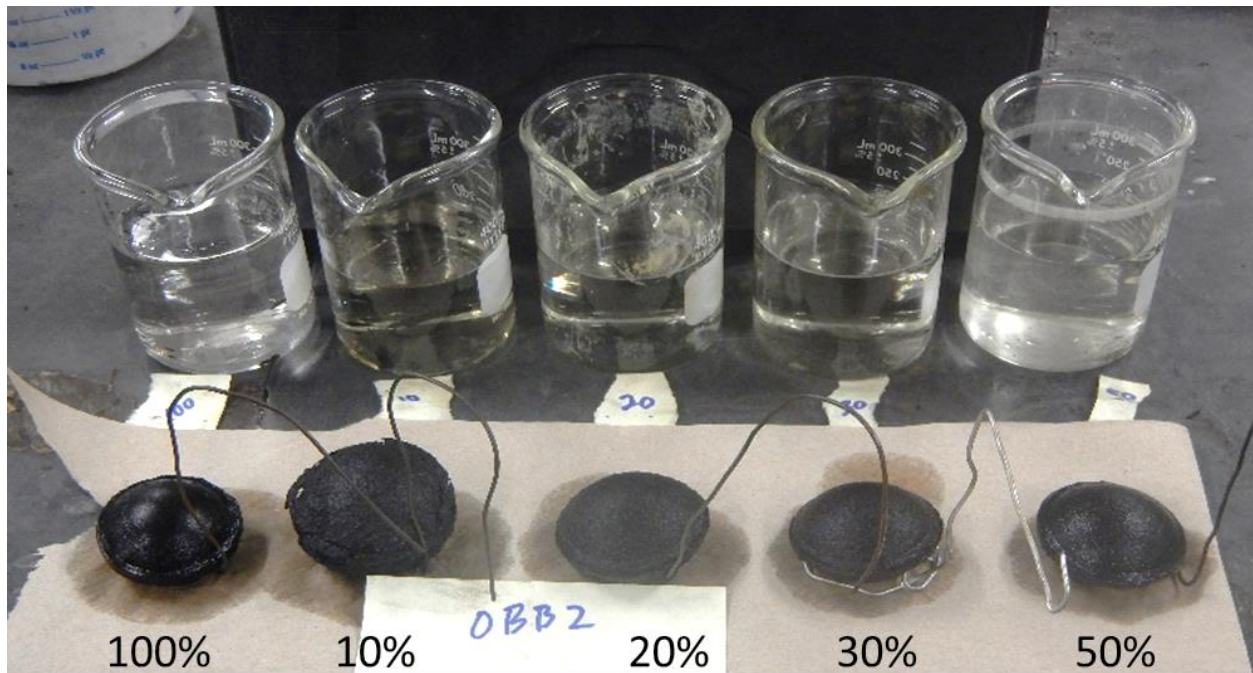
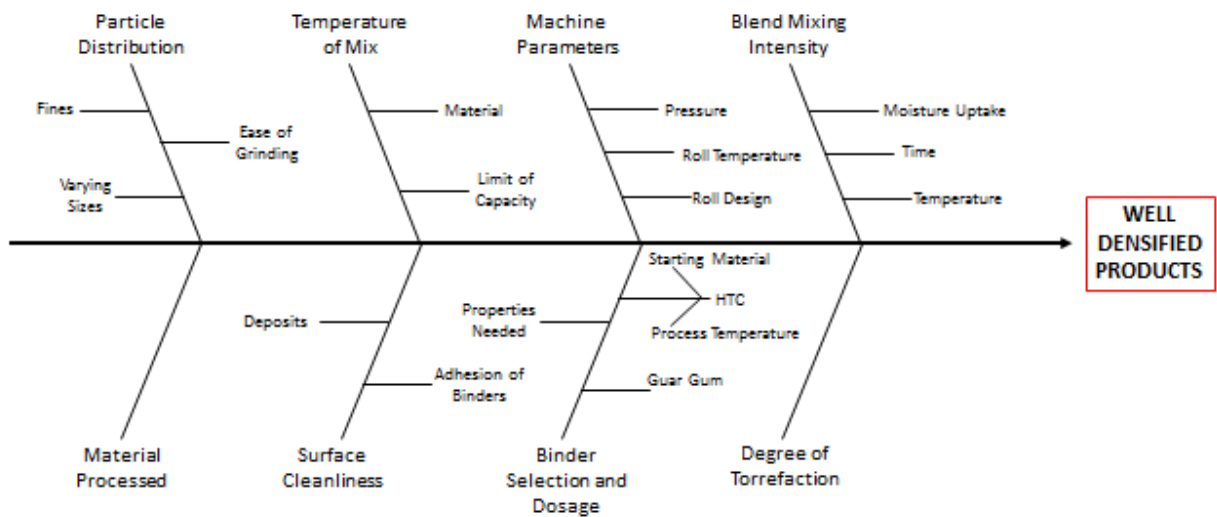


Figure I-20: Inclusion of HTC processed outer birch bark in torrefied blends and moisture resistance.

Effective Densification Requires Manipulation of Many Variables

The scoping studies have shown that the densification process is not simple. It is highly dependent on a number of conditions that have to be carefully manipulated. The conditions vary by the degree of torrefaction employed, the type of binder used, the size consist of the particulate assemblage, the temperature of mix preparation, the starting material used in torrefaction, the imbibed moisture content, the use of hydrothermally carbonized materials in the production of fuel products, and the type of densification equipment used to produce the densified compacts. The conditions employed on the densification machines are also a key factors in gaining high quality products. For the example of briquetting, the variables of importance are illustrated in Figure I-21.

Key Variables for Acceptable Briquettes



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Figure I-21: Key variables for forming acceptable briquettes.

SECTION II

Investigation of Physical Properties of Densified Products

The following is a summary of the mechanical quality of various forms of briquettes made using briquetting equipment supplied by K.R. Komarek, Inc. in comparison to consumer charcoal and industrial standard Powder River Basin (PRB) coal. All the briquettes produced at the Renewable Energy Lab (REL) in NRRI's Coleraine facility used the same die volume and geometry. The physical tests included crush strength to measure the point at which the briquettes fracture at a constant cross head speed of 1 mm/min (0.041 in/min), tumbling durability using the Kansas State Tumbling Procedure [<https://www.bookstore.ksre.ksu.edu/pubs/MF3228.pdf>], drop durability from 0.914 m (36 inches), and moisture uptake using a standard 24-hour immersion period. To date, the variables to be considered when analyzing the results are as follows: die geometry size, roll speed, feed speed, grind spec of the torrefied material, moisture content of post-treated feed stock, variability from estimating total mass of torrefied wood and adding the appropriate amount of binder, steam/heat blanket, species of wood used for torrefaction, type of binder, outlet and ejection temperature, and die temperature. This is not an exhaustive list of variables. Feedstock used for this portion of the studies undertaken consisted of red oak and pine torrefied at the REL-Coleraine.

Pre-Treatment Protocol

Hot Briquetting

The pretreatment technique used in the briquetting tests included both hot and cold compaction. With hot compaction, the material exiting the kiln is cooled in a cooling screw with indirect water from a nominal temperature range of 299°C – 316°C (570°F – 600°F) down to 93°C (200°F) before it enters a bucket elevator and surge bin. (This cooling is necessary because of bucket elevator belting temperature limitations.) The surge bin then releases the material into a turbulizer, where it is ground to a typical size specification of 60–70% passing 30 mesh (0.595 mm) with the balance passing an 8 mesh (2.36 mm). The turbulizer transfers approximately 16.7°C (30°F) of frictional heat to the material. The material then enters a 1.415 m³ (50 ft³) triple pass ribbon blender equipped with live 103.4 kPa (15 psi) steam injection and insulated heating blankets. Liquid or powder-type binder is added at this point with live steam addition for approximately 15 minutes. At this point, the moisture level of the material is brought to approximately 4–10%, with the temperature being approximately 93°C (200°F). The material is then released batchwise and fed manually into a Komarek B220 rotary briquette machine. With this technique, the steady state outlet temperature of the briquettes is maintained nominally in the range of 71°C – 79°C (160°F – 175°F). See Figure II- and Figure II-2 for the basic production circuit.

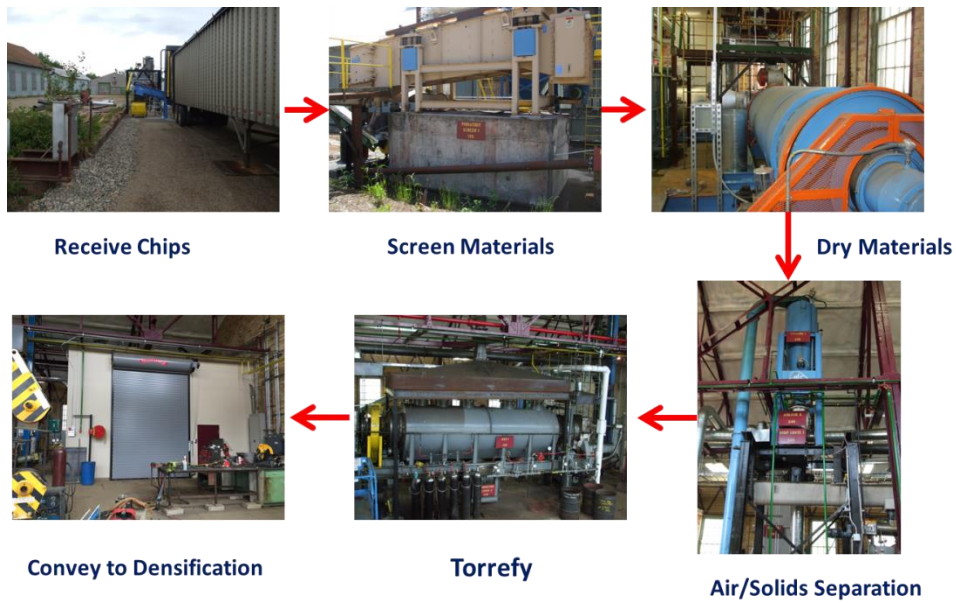


Figure II-1: Basic production circuit used to produce torrefied fuels at NRRl’s Renewable Energy Laboratory.

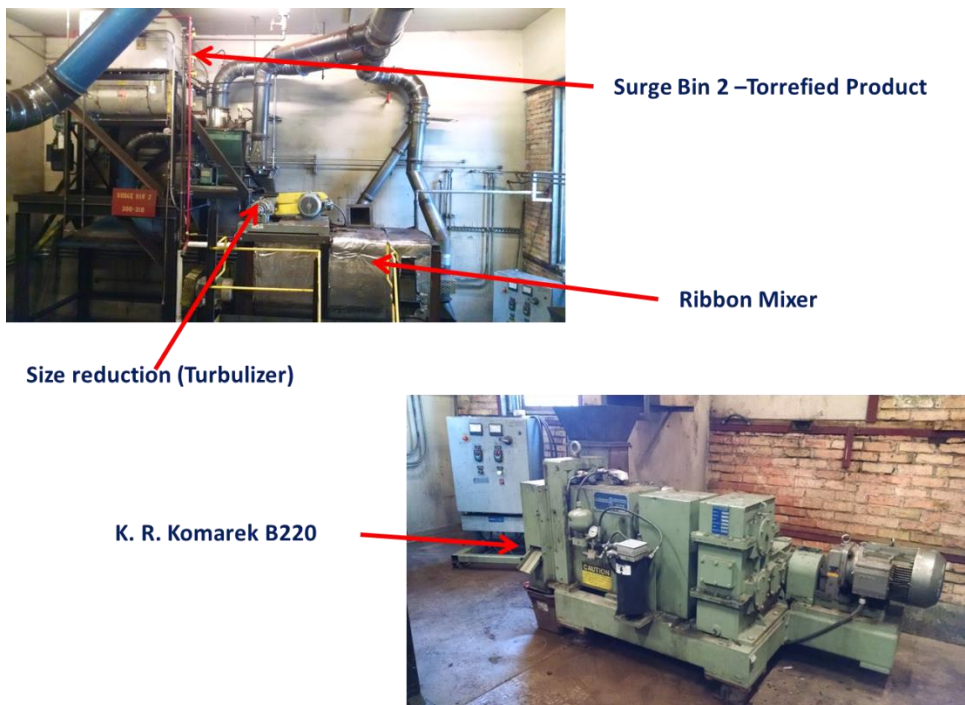


Figure II-2: Basic densification circuit used to produce torrefied fuels at NRRl’s Renewable Energy Laboratory.

Cold Briquetting

The cold-forming technique used ambient cooled material that was collected in 55-gallon steel drums and set aside. The material had aged in the drums for approximately 2–3 weeks before being batch fed into a surge bin upstream of the turbulizer. The surge bin then released material to the turbulizer, where it picked up about 16.7°C (30°F) of frictional heat before being released to the mixer. Binder was added at this point and allowed to mix for approximately 15 minutes. The material exiting the mixer was approximately 15.6°C – 21°C (60°F – 70°F) and then fed batchwise to the Komarek B220 briquette machine. With this technique, the steady state outlet temperature of the briquettes is maintained nominally in the range of 32°C – 49°C (90°F to 120°F).

Binders

All binders were tested at nominal 1% inclusion based on the dry weight of the estimated mass in the mixer. The binders tested included guar gum, tapioca starch, RW6900 starch, and PVA. In addition, runs were completed with both hot and cold briquetting with no binder across pine and red oak species.

Procedure

Drop Durability

Drop durability is performed by dropping the briquette from 0.914 m (36 inches) onto a plate 20 times or until failure (Figure II-3). The initial mass is first determined, and the material is then dropped 20 times or until failure and the final mass measured. Mass is measured using an OHAUS gram scale: Model AR5120. Percent survival is determined by the following calculation: $\left(\frac{\text{Final Mass After 20 Drops}}{\text{Initial Mass}}\right) * 100$.



Moisture Uptake

Moisture uptake is determined by completely submerging the briquette in water using a 400 mL beaker (Figure II-4). The initial mass of the briquette is determined (OHAUS gram scale: AR5120). The briquette is placed in a metal basket to facilitate removal from the beaker. Briquettes are removed from the beaker and placed on a paper towel, where the surface moisture wicks off for 2–3 minutes. The mass is then determined. This is done at time points: 15 minutes, 1 hour, 3 hours, and 24 hours. A decrease in mass at longer time periods corresponds to substantial disintegration of the briquette. Percent moisture uptake was determined at each time point using the following calculation: $\left(\frac{Final\ Mass - Initial\ Mass}{Initial}\right) * 100$.

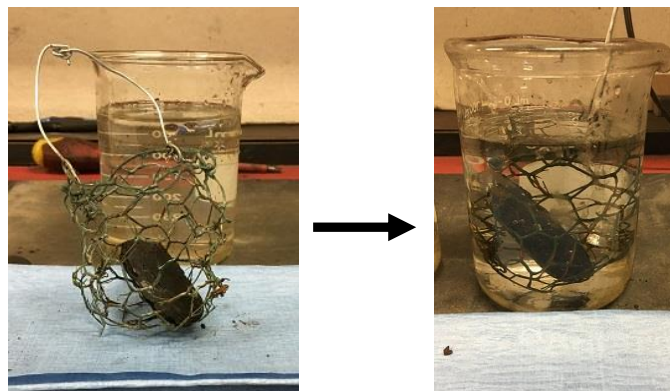


Figure II-4: Moisture uptake illustration.

Particle Distribution

Particle distribution is determined using 10 (2.0 mm), 20 (0.841 mm), 30 (0.595 mm), 50 (0.297 mm), and 100 (0.149 mm) mesh sieves shaken for five minutes using a Ro-Tap sieve shaker (Model: RX-29) (Figure II-5). After five minutes, the particle mass at each mesh size is determined using an OHAUS gram scale (Model AR5120) and recorded.



Figure II-5: Determining particulate size distribution.

Kansas Durability

Tumbling durability is performed using a Kansas State Tumbling Can with a modified approach to accommodate briquettes versus pellets (Figure II-6). The diameter of 10 briquettes is measured, and the average is used to determine the appropriate screen size: 11.2 mm (7/16-inch mesh). Six hundred grams of each sample are measured out and tumbled for two minutes at the standard rate of 60 revolutions per minute to eliminate the flashing around the briquette. After the two-minute conditioning, the briquettes are removed, sieved using the 11.2 mm (7/16-inch) mesh on the RoTap for two minutes, and 500 grams of briquettes are measured. The 500 grams (1.1 lb) are then placed back into the tumbling can and tumbled for 10 minutes at the standard 60 rotations per minute. After 10 minutes, the sample is removed, placed on the 11.2 mm (7/16-inch mesh) sieve, sieved for 2 minutes, and the +11.2 mm (+7/16-inch mesh) sample is weighed. Percent durability is determined using the following formula:

$$\left(\frac{\text{Final Mass}}{500}\right)*100.$$



Figure II-6: Kansas durability illustration.

Crush Strength

The crush strength of the briquettes is measured using an ATS tensile and compression testing machine. The Janka ball is attached to the screw attachment, as shown in the left-hand side of Figure II-7, and the calibration checked. Individual briquettes are placed on the steel plate, and the Janka ball is positioned just above, and directly centered over, the briquette. The Janka ball comes down towards the briquette at a crosshead rate of 1 mm/min (0.041 in/min) until fracture, measured in pounds force (4.448 N = 1 lb_f).

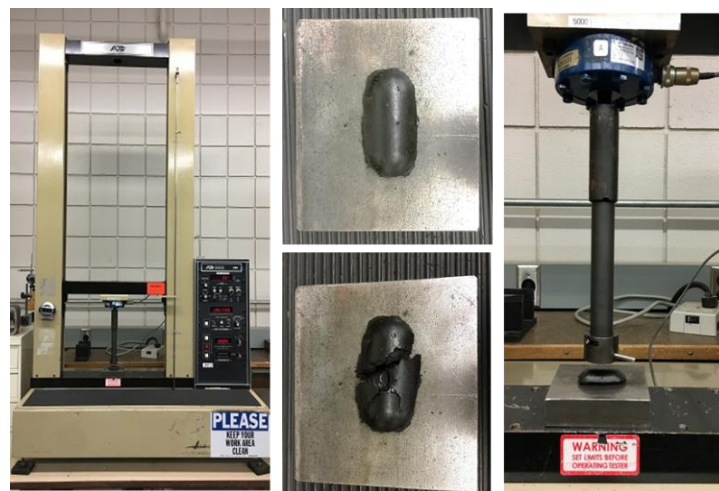


Figure II-7: Crushing strength test configuration.

Moisture Penetration Index (MPI)

The moisture penetration index measures the amount of time it takes for a droplet of water to penetrate the surface of a puck, briquette, or pellet. A needle free syringe is filled with water and a single drop is placed on the surface of the sample (Figure II-8). At the same time, a droplet is placed on the surface, a timer is started and monitored until the droplet completely penetrates the surface of the sample. Time is measured, as well as physical characteristics: a protruding surface after penetration, signs of disintegration, or no change at all. This test is done before the 24-hour moisture uptake to quickly evaluate and estimate the moisture-resistance properties of the sample. In general, those samples that have MPIs > 60 minutes will likely survive direct immersion in water for 24 hrs.



Figure II-8: Moisture penetration illustration.

Microwave Steam Production

This pre-treatment procedure is used to emulate steam production to activate lignin and other binding molecules within the dry sample (Figure I-9). The moisture content of the sample is determined using an OHAUS Moisture Balance (MB200). The moisture content is then adjusted to 20–30% depending on the material consistency. The target moisture content is 8–12%; the moisture is adjusted to 20–30% initially to allow for steam production during the microwave process. The final mass of the sample is calculated based on the target moisture content. Approximately 40 grams of sample is placed in a 473 mL (16 oz) plastic container, weighed initially, microwaved at 30- to 60-minute intervals, and weighed after each interval until 8–12% moisture content and 88°C – 99°C (190°F – 210°F) sample temperature is achieved. This sample is loaded into the forming dies on the puck press to make round briquettes.



Figure II-9: Microwave heating of blend to correct moisture level.

Hydraulic Press

The initial moisture content of the torrefied wood mixture was determined using an Ohaus moisture balance (Model: MB200), then adjusted to 27–30% moisture. Approximately 40 grams of milled sample is used to make each briquette and is measured in a 473 mL (16 oz) plastic container using an OHAUS gram scale (Model: AR5120). The mixture is then heated using the “microwave steam production” procedure until a final target moisture content of 8–12% is achieved. Briquettes are formed using room temperature dies, or those heated to 260°C (Thermolyne 48000 furnace), pressed at 28,959 kPag (4,200 psig) for 60 seconds, ejected at a temperature between 190°C – 210°C, and cured overnight. A hydraulic press (Enerpac: RR-5013) is used to densify the briquette. The pressure applied to the briquette is determined using *Calculation 1* below. The pounds per square inch, gage (4200 psig) is multiplied by the ram area, and this product is divided by the contact area to get a pound-per-square-inch reading. This pressure is a starting parameter that we try to emulate during rotary compaction once the experiment is scaled up (Figure II-10).

Calculation 1:

$$\left(\frac{(\text{psig})(\text{Ram Area } \text{in}^2)}{\text{Contact Area } \text{in}^2} = \text{psi} \longrightarrow \frac{4200 \text{ psig} (11.06 \text{ in}^2)}{3.14 \text{ in}^2} = 14793 \text{ psi} \right)$$

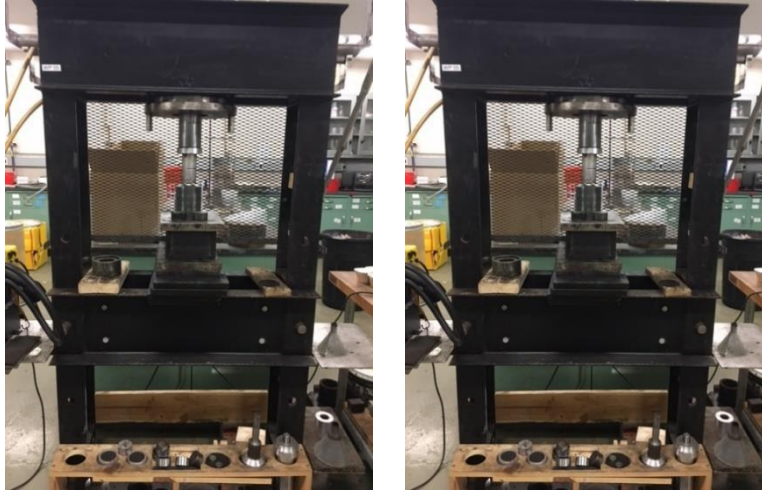


Figure II-10: Briquetting of prepared mixture.

Results

During the investigation, various blends of material were characterized using the systems noted in the previous sections. Those results are summarized in this section. The formulations investigated are summarized in Table II-1.

Table II-1: Formulations tested.*

Consumer Charcoal (nominal ~2-inch size)	1% Guar: 99% Oak Hot
Industrial PRB Coal Chunks (~2-inch size)	1% Guar: 99% Pine Hot
100% Oak Hot	1% PVA: 99% Oak Hot
100% Oak Cold	1% RW6900: 99% Oak Hot
100% Pine Hot	1% Tapioca: 99% Oak (1700 psi, Hot)
100% Pine Cold	1% Tapioca: 99% Oak (2000 psi, Hot)
1% Guar: 49.5% PRB Coal: 49.5% Oak	

*psig 2700 to 3000 on briquette rolls unless otherwise noted (1 smooth roll: 1 pocketed roll, 1-inch x 2-inch briquette (25.4 mm x 50.8 mm))

Figure II-11 illustrates the results for crush strength. Briquettes of 100% pine formed under either hot (steam) or cold (no steam) conditions had the highest crush strength and required over 300 pounds of force to fracture. 100% oak hot, 100% pine hot and cold, and consumer charcoal outperformed industrial coal.

Figure II-12 shows the results for the Kansas State durability tests. Consumer charcoal had the highest survival rating. 100% oak hot, 1% guar: 99% oak, 1% PVA: 99% oak, 1% RW6900: 99% oak, and 1% tapioca: 99% oak at 2,000 psi had greater survival performance than industrial coal.

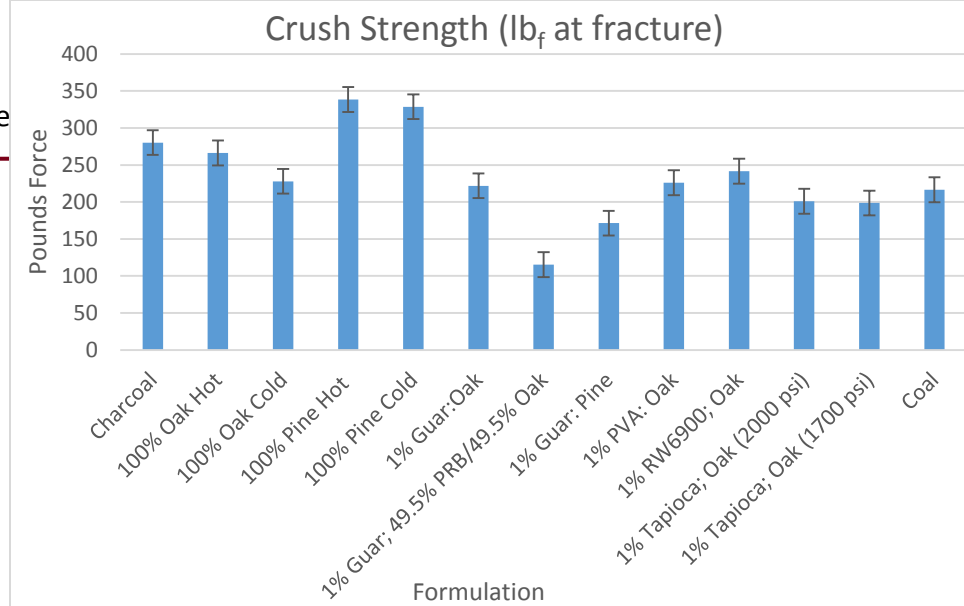


Figure II-11: Average crush strength in pounds force at fracture.

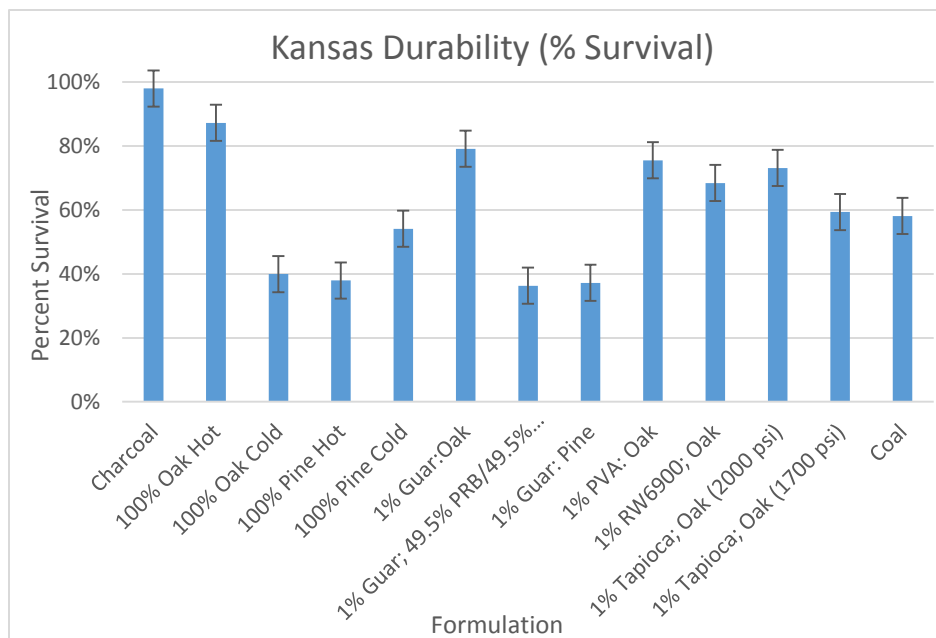


Figure II-12: Tumbling durability survival of 500 grams of each sample using the Kansas State Tumbling Can.

The results for drop durability are shown in Figure II-13. Consumer charcoal and 100% hot oak had no significant difference in survival between 5 and 20 drops. All other formulations had a significantly lower survival at 20 drops compared to 5 drops. Most blends of the torrefied fuels had better drop durability compared to sub-bituminous commercial coal but not quite as good as commercially produced charcoal, which uses significant amounts of binder above those used for the torrefied materials.

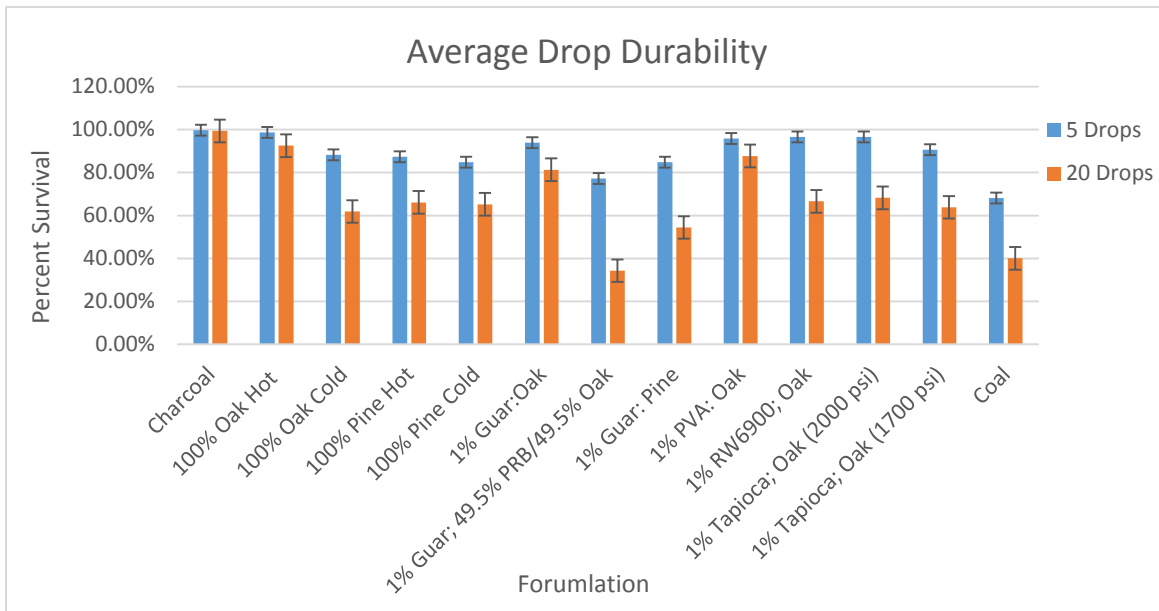


Figure II-13: Average drop durability after 5 drops and 20 drops.

Figure II-14 illustrates the 24-hour moisture uptake by different blends of materials for the briquettes processed. Red bars indicate that the briquette disintegrated, and thus, did not survive the full 24 hours. Industrial coal and 100% oak hot briquettes demonstrate a significantly lower percentage of moisture uptake compared to the rest of the formulations. Briquettes formulated using torrefied oak and a binder had a significantly lower percentage of moisture uptake than briquettes formulated using torrefied pine.

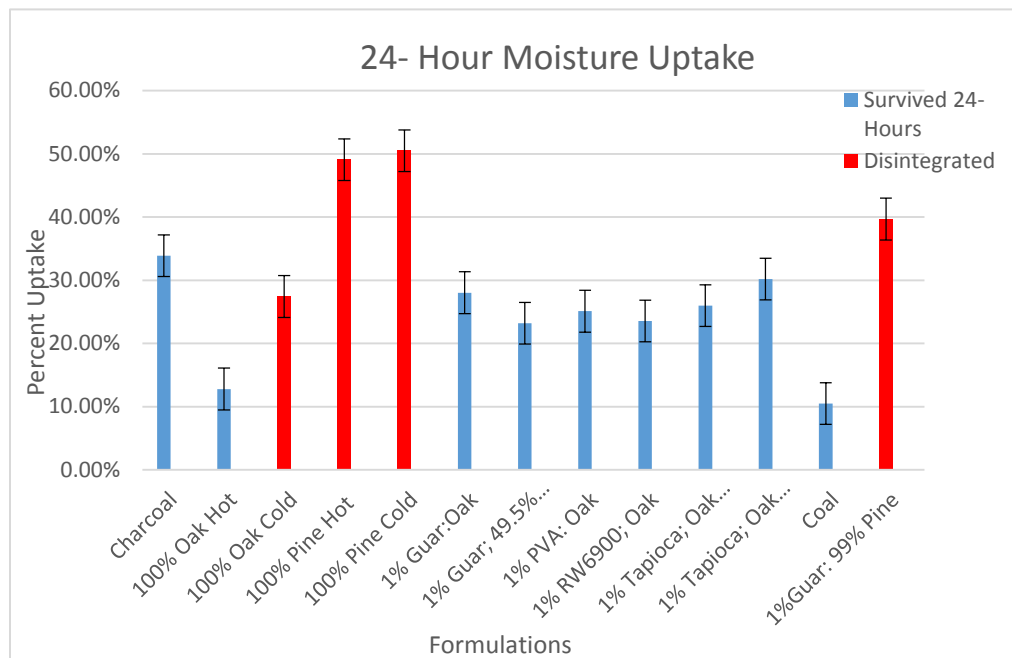


Figure II-14: Greatest percentage of moisture uptake over a 24-hour immersion period.

Briquette Aging Results – 5 Day Cure vs 30 Day Cure

The drop durability data is illustrated in Figure II-15. The data has substantial variation. 100% oak hot, after 20 drops, has significantly less durability after aging for 30 days. At 20 drops, 1% guar/ 49.5% PRB coal/ 49.5% oak has significantly increased survival after aging for 30 days. There are no significant differences between 5 days and 30 days after only 5 drops. In general, the materials possessed sufficient strength to survive sufficient material handling and transportation requirements compared to coal.

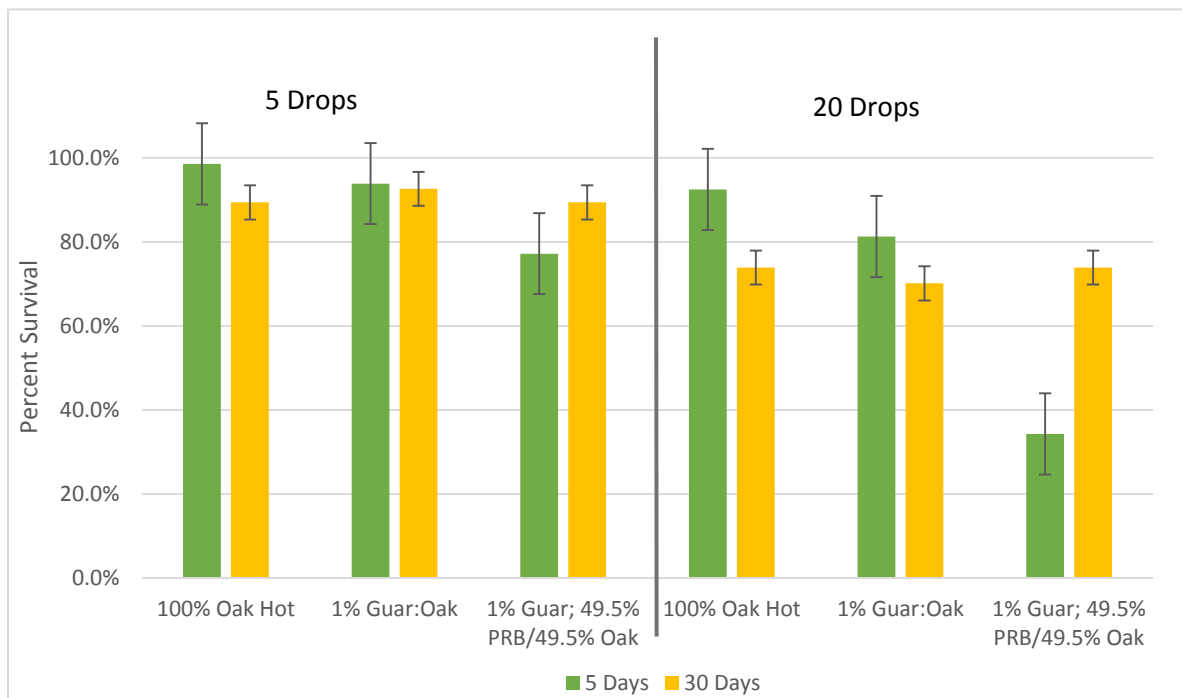


Figure II-15: Average drop durability for samples at 5 days versus 30 days measured after 5 drops and 20 drops.

Figure II-16 compares Kansas Durability for some select mixes. There is no correlation in this data set that suggests briquettes have increased survival after aging for 30 days. Overall 100% oak hot has the greatest survival.

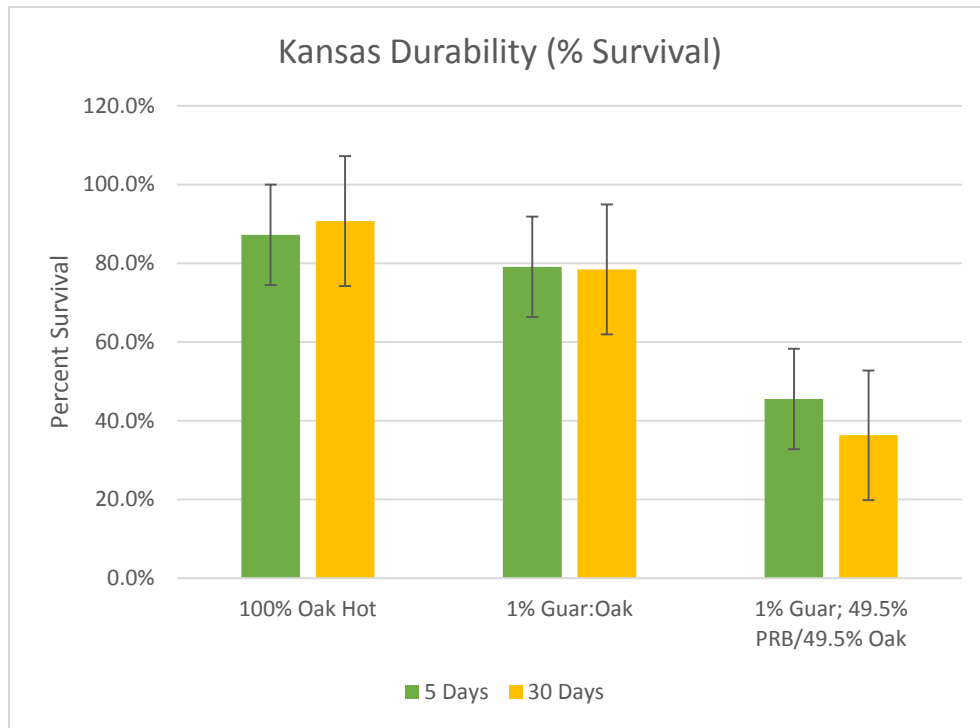


Figure II-16: Comparison of Kansas Tumbling Durability for briquettes aged 5 days and 30 days.

There is no significant increase in crush strength required to fracture after aging briquettes for 30 days as shown in Figure II-17. The crushing strength obtained is sufficient for normal handling and transportation and storage purposes.

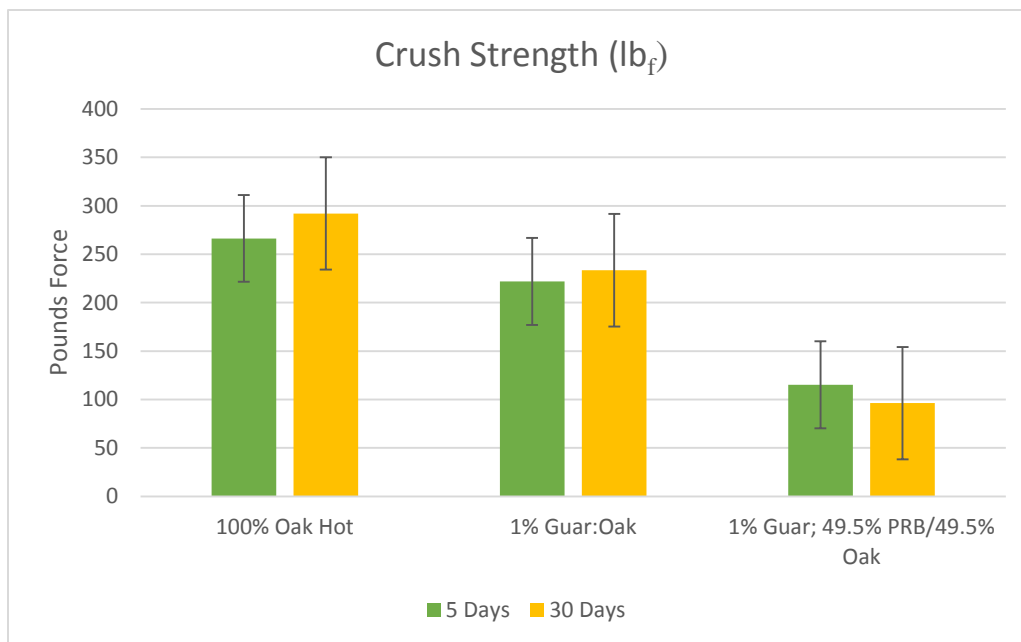


Figure II-17: Comparison of Crush Strength in pounds force for briquettes aged 5 days and 30 days.

Figure II-18 compares briquette data after curing for 5 days versus 30 days for moisture uptake. Our target for this measure is to be less than 30%. Briquette blends for 1% guar: 99% oak and 1% guar: 49.5% PRB coal: 49.5% oak briquettes had notably more moisture uptake after curing for 30 days. In general, the moisture uptake after five days is better or near the target value of less than 30% and shows that there is potential for meeting this desired property.

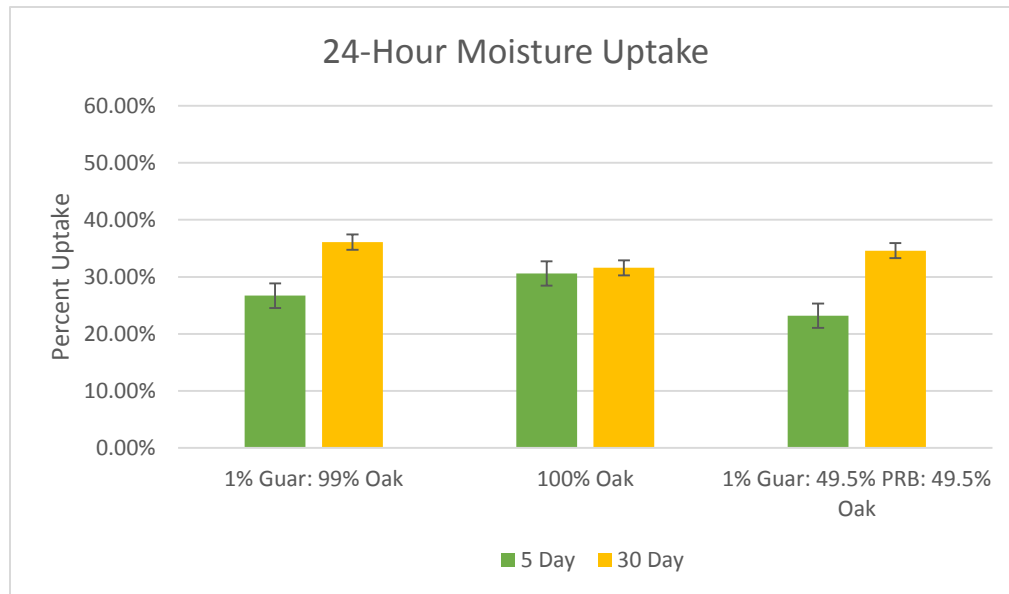


Figure II-18: Greatest percentage of moisture uptake over a 24-hour immersion period.

Figure II-19 illustrates the particle distribution of torrefied oak versus torrefied pine. Both oak and pine had the greatest distribution of fines -0.149 mm (-100 mesh) and greater than 60% passing -0.595 mm (-30 mesh). Pine had a greater percentage of -30 mesh than that of oak and, as a result, had overall slightly larger amounts of fine materials.

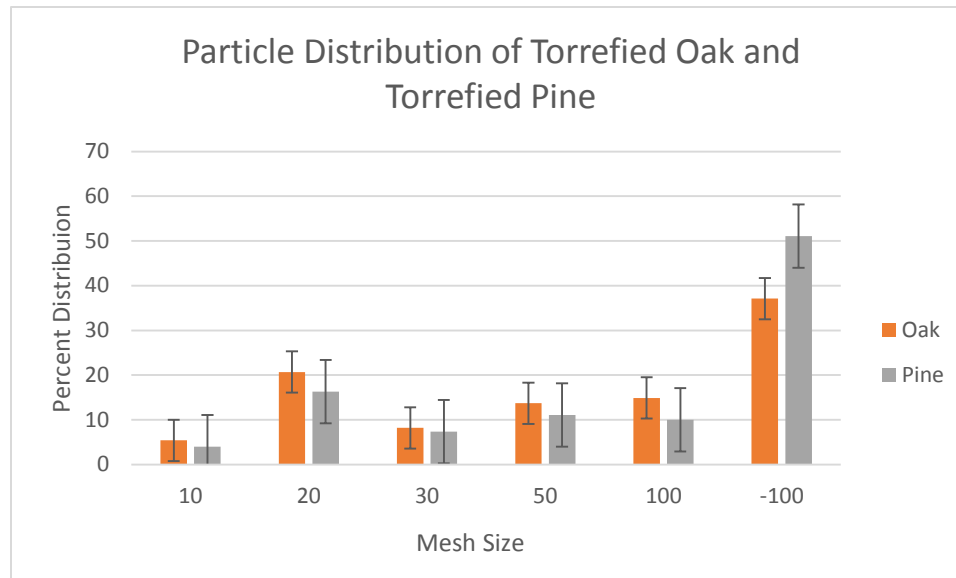


Figure II-19: Particle distribution of oak versus pine after torrefaction

Overall Property Evaluation for the Various Blends Tested

The results for the various blends are shown in summary form in Table II-2 for the various tests that have been reviewed. Overall, commercial charcoal that contains a high level of binder performed very well in the tests, but various blends of torrefied materials also performed well. Our experience in comparing the press made briquettes versus those on a commercial briquetting machine indicate that the commercial conditions will likely result in improved properties for most categories, but as can be seen, satisfactory results can be attained even for certain blends made using the laboratory press.

Discussion

In overall performance, there were several formulations of briquettes from torrefied wood that outperformed currently used industrial coal: which rated only as satisfactory for overall mechanical testing performance. The NRRI formulations that outperformed industrial coal overall include: 100% oak hot, 100% pine hot, 1% guar: 99% oak steam; 1% RW6900: 99% oak steam. The rating scale used gave each formulation a number 1–12, unless a tie was noted, in each mechanical test—tumbling durability, drop durability survival after 5 drops, drop durability after 20 drops, and crush strength—and a rating of 1–3 for percent moisture uptake: (1) < 30% uptake; (2) > 30% uptake; (3) > 30% uptake and disintegrated before 24-hour period. Each score was added, and ratings were assigned as seen in Table II-2.

Figure II-11 graphically represents overall crush strength where the Janka Ball is impinged into a briquette with a cross head speed of 1 mm/min (0.041 in/min). Briquettes that performed the best and required the most force to fracture are 100% pine hot and cold forms. This is likely due to the more fibrous nature of pine when compared to red oak. Consumer charcoal and 100% oak hot had a crush strength > 1,112 N (250 lbf). Most of the formulations were comparable to industrial coal, with a load with crush strengths below 1,112 N (250 lbf).

Figure II-12 graphically represents tumbling durability using the Kansas State tumbling can and tumbling procedure at 60 revolutions per minute for 10 minutes. Charcoal and 100% oak hot had the greatest survival rate at > 80%; 1% guar: 99% oak hot, 1% PVA: 99% oak, 1%

RW6900: 99% oak, and 1% tapioca: oak (2,000 psi) had survival ratings > 60%. All other formulations, including industrial coal, were at ratings < 60% survival.

Figure II-13 graphically represents average drop durability that was measured after both 5 drops and 20 drops. Consumer charcoal had 99% survival after both 5 and 20 drops; the comparable NRRRI briquette 100% oak hot, had 98% after 5 drops and 92.5% after 20 drops. Briquettes that had greater than 90% survival after 5 drops include: 1% guar: 99% oak, 1% guar: 49.5% PRB: 49.5% oak, 1% guar: 99% pine, 1% PVA: 99% oak, 1% RW6900; 99% oak, 1% tapioca: 99% oak (2000 psi), and 1% tapioca; 99% oak (1700 psi). All other formulations had < 89% survival after 5 drops. After 20 drops, consumer charcoal and 100% oak hot had > 90% survival: all other formulations had < 89% survival, generally falling in the 50–60% range.

Figure II-14 graphically represents the percent of moisture uptake after 24 hours of water immersion. Orange bars indicate that the briquette had significant disintegration and did not survive the 24-hour period. Ideally, the briquette would have a moisture uptake of < 30%. Coal and 100% oak hot had the lowest percent of moisture uptake < 15%. Consumer charcoal had > 30%, and all other surviving formulations fell in the range 16% > 29.9%. The briquettes that did not survive included 100% oak cold, 100% pine hot, 100% pine cold, 1% guar: 99% pine.

Table II-2: Summary Table of NRRI Formulation Performance to Consumer Charcoal and Industry Standard Coal (Five-Day Cure)

Formulation	Kansas Durability	Drop Durability – 5 Drops	Moisture Resistance	Crush Strength (lb _f)	Overall
Consumer Charcoal					
100% Torrefied Oak Steam					
100% Torrefied Oak No Steam					
100% Torrefied Pine Steam					
100% Torrefied Pine No Steam					
1% Guar: 99% Torrefied Oak Steam					
1% Guar: 99% Torrefied Pine No Steam					
1% PVA: 99% Torrefied Oak Steam					
1% RW6900: 99% Torrefied Oak Steam					
1% Tapioca (2000 psi): 99% Torrefied Oak Steam					
1% Tapioca (1700 psi): 99% Torrefied Oak Steam					
1% Guar: 49.5% PRB Coal: 49.5% Torrefied Oak					
Industrial Coal					

Rating Scale:

Kansas Durability and Drop Durability (% Survival)

Excellent >85% 	Satisfactory 84>75% 	Poor <74%
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Mois

Overall

Excellent 10-19 	Satisfactory 20-29 	Poor >29
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Moisture Resistance

Excellent <30% 	Satisfactory >30% 	Disintegration
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Crush Strength (lb_f)

Excellent 400>280 	Satisfactory 279>150 	Poor >149
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Figure II-15 graphically represents average drop durability after 5 drops and 20 drops. This graph compares briquettes aged 5 days and 30 days. There is variation among the data, not leading to any conclusive correlations or trends. The formulation that had greater survival after curing for 30 days is 1% guar: 49.5% oak: 49.5% PRB coal.

Figure II-16 graphically represents tumbling durability survival for formulations after curing for 5 days and 30 days. No significant improvement was noted for briquettes aged 30 days. 100% oak hot was the only formulation that remained above 80%.

Figure II-17 graphically represents crush strength in pounds force required to fracture. No significant change was noted between 5-days-old and 30-days-old samples. Formulations that had slight improvement after 30 days included 100% oak hot and 1% guar: 99% oak. 1% guar: 49.5% PRB: 49.5% oak had a slight decrease in strength after 30 days.

Figure II-18 graphically represents the greatest percentage of moisture uptake over a 24-hour period. All the briquettes survived the 24-hour immersion, both at 5-day cure and 30-day cure. Samples that had a slight increase in moisture uptake after being allowed to cure for 30 days included: 1% guar: 99% oak and 1% guar: 49.5% PRB coal: 49.5% oak. Little to no change was observed for 100% oak.

Particle Distribution

Figure II-19 graphically compares the particle distribution of turbulized torrefied material. Ideally, material will have a greater-than-60% distribution at passing 30 mesh (0.595 mm), which was achieved in this experiment. Oak had 65.7% passing 30 mesh (0.595 mm), while pine had 72.3% passing. Oak had greater percentages of distribution at 10, 20, 30, 50, and 100 mesh, indicating that it has a coarser and fibrous grind specification. Pine had the greatest number of fines passing 100 mesh.

Based on the results from this bench scale work, blends were identified for further testing on the B220 briquetting circuit. Barrel quantities of these blends were then produced from 100% biomass with guar gum binder and a 50:50 blend of torrefied biomass/fossil coal with guar binder. These materials were then tested on a stationary hearth in two one-quarter-scale steam engines, and the results will be reviewed in the next section of the report.

Bulk Density

Tests were run on select blends of biomass materials on the commercial scale B220 from Komarek. The bulk density of the formed briquettes using the 25.4 mm x 50.8 mm (1 in x 2 in) die with one smooth roll and one pocketed roll was nominal, 592 kg/m³ – 624 kg/m³ (37 – 39 lb/ft³). Operation with one smooth roll and one pocketed roll eliminated splitting that was evident with dual pocketed rolls. It should be mentioned that operation with a smaller die 19.05 mm x 44.5 mm (nominal 0.75 in x 1.75 in) raised the bulk density to nominal 656 kg/m³ (41 lb/ft³). Logistical shipping advantages become more evident as bulk density surpasses 640 kg/m³ (40 lb/ft³). It is envisioned that a triangular- or pyramid-shaped die may offer more densification. This die shape requires further discussion with the die manufacturer.

Conclusions

- Excellent briquettes with good physical properties could be made from well-torrefied materials using the briquetting practices developed.
- Torrefied materials can possess better physical properties than that for industrial coal.

-
- Weathering resistance can be attained with the right selection of binder and raw material using the briquetting practices employed.
 - The type of starting raw material can have an influence on the final product properties and likely will require tuning of the whole production system to the input materials employed from torrefaction through final densification.
 - The use of one pocketed roll and one smooth roll produced biofuel briquettes with no tendency for splitting, and this configuration may be desirable for actual commercial production.
 - The employment of higher densification pressures combined with an integrated production system of torrefied biofuel production demonstrates that high quality fuel products can be achieved.
 - If binders are required, satisfactory products can be made at low binder levels if properly prepared mixes are produced through grinding and mix preparation.
 - Final product attributes meet or exceed that for sub-bituminous coal, and the fuel values attained for the torrefied products match or exceed that possessed by charcoal or coal.
 - Further improvement is likely as higher pressures, improved geometries, and tuned system conditions are employed.
 - It is likely that the parameters identified in this work can be duplicated at the commercial level and lead to viable commercialization of these fuel products.

SECTION III

Use of Briquette Blends in Stationary Hearths of Two One-Quarter-Scale Steam Locomotives at the Milwaukee Zoo

As noted in the previous sections, various techniques were used to evaluate creation of densified fuel materials under various production and test conditions. Usage of these materials requires an intimate knowledge of the fuel processing systems that will be employed in storing, handling, comminuting, and combusting the fuel products. During the CAWES program, various tests have been summarized in using commercially produced pellets in powder coal injection (PCI) systems. The largest test was conducted at Portland General Electric's Boardman facility, and this test ultimately consumed close to 5,000 tons of torrefied pellets. Only minor modifications were needed to accommodate this fuel use, and up to 100% substitution for sub-bituminous coal was achieved. Other tests were held at Minnesota Power's Taconite Harbor plant and Gulf Power's Plant Scholz. In all cases, it was demonstrated that substantial substitution could be achieved in using the biomass-derived fuel in replacing the commercial coals typically used as noted; the tests at the three power plants used PCI systems that required the pellets to be ground using large pulverization systems.

However, other significant coal-based fuel systems do not pulverize the coal before it is combusted to provide heat for boiler or industrial use. This is often achieved by using stoker systems to feed larger pieces of coal onto a grate, where it then burns and provides hot flue gases that are used to heat pipes to generate steam for various purposes. As part of the program, investigations on the use of densified briquettes that more closely simulate this type of stationary hearth were undertaken. These tests were conducted at the Milwaukee Zoo using two one-quarter-scale steam engines that traverse a 1.1-mile oval and haul passengers around the zoo during normal operations at the facility.

The scale of these engines is shown in Figure III-1 and Figure III-2. As can be seen, even at this scale, the equipment is significant. The engines provided a very good test bed for determining the suitability of using torrefied fuels as a direct substitute for the coals typically used at the Zoo. Zoo management desired to see if the coal could be replaced, due to the need to enhance environmental performance including stack emissions and opacity and to develop a direct substitute for coals that are becoming more difficult for the Zoo to obtain. Three test periods took place using the fuels produced by NRRI. In the first case, ram briquettes were produced using the B-100 machine shown in Figure I-6 and illustrated in Figure III-3. In the second case, torrefied pellets were employed and supplied from New Biomass Energy. In the third case, two blends of materials were employed: (1) 100% torrefied woody biomass, and (2) a 50:50% blend of sub-bituminous coal and torrefied woody biomass. For the woody torrefied biomass, briquettes were made with and without binders. The blend with coal used a binder. The binder employed was guar gum. In the third test period—the densification system is illustrated in Figure II-2—the briquettes were made using one pocketed roll and one smooth roll based on previous trials using various roll configurations. The combination of one smooth and one pocketed roll gave excellent densification from the outside to the interior of the briquette. The materials produced using this system is shown in Figure III-4. A comparison of the fuel briquettes to the typical coal size employed is shown in Figure III-5.



Figure III-1: Steam engines used during the stationary hearth test programs at the Milwaukee Zoo.



Figure III-2: Steam engines heating up boilers using typical coal fuels.



Figure III-3: Briquettes supplied for first tests at the Milwaukee Zoo.



100% Torrefied Biomass



50% TB:50%PRBCoal

Figure III-4: Briquettes used during the second test at the Milwaukee Zoo.

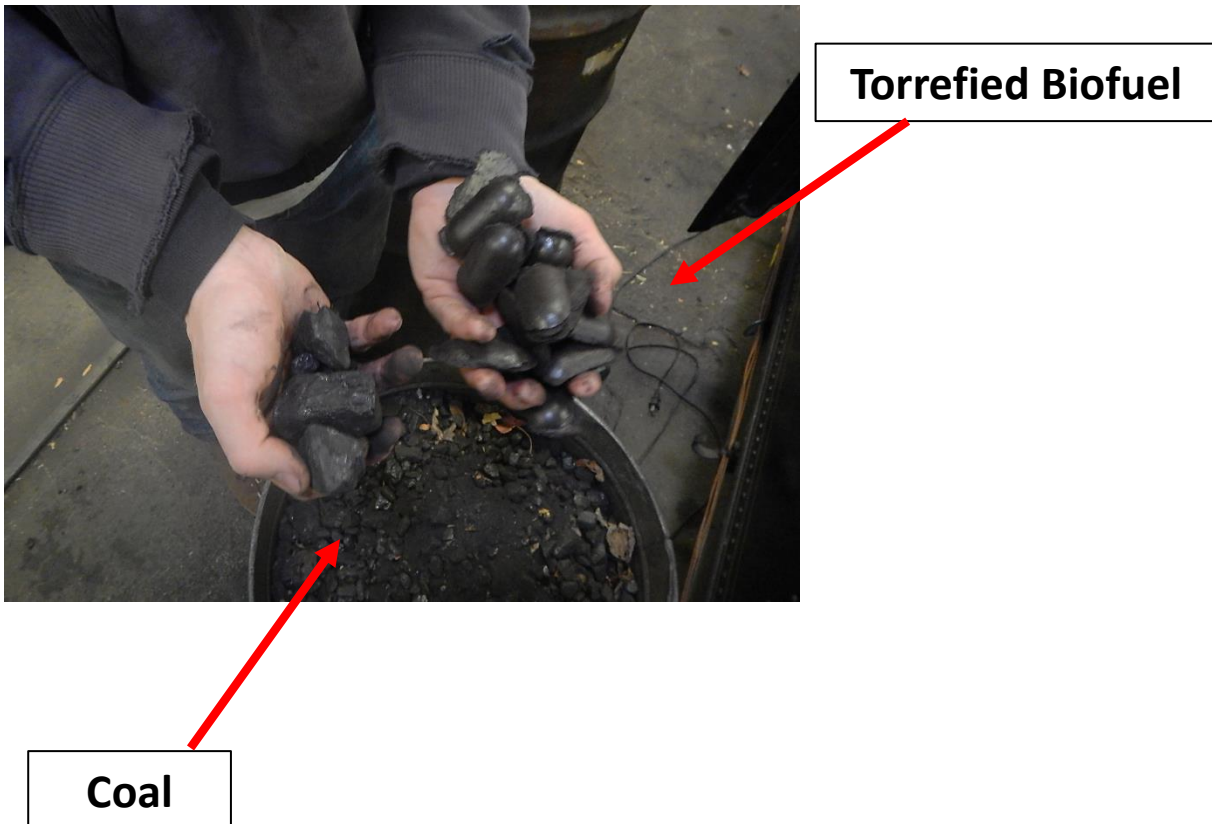


Figure III-5: Comparison of fuel briquettes and coal used to power steam locomotives.

Various criteria were established by the zoo train operators to gauge the potential utility of the fuel, and these parameters were: (1) safe operation of the locomotive without sparklers emitting from the locomotive stack; (2) full bed coverage of the grate in order to protect the grate bars; (3) improved emissions, including opacity and reduced sulfur emission from the locomotive stack; (4) equivalent of better fuel content compared to the coals typically used; (5) good flame characteristics to ensure efficient heat transfer to the boiler tubes; and (6) enough energy to allow the engines to perform their function of pulling a fully loaded zoo train around the zoo.

The results from the first trial using the B-100 ram briquetter were poor, as when the train was fully engaged in pulling its load, the draft on the combustion system caused sparklers to be emitted from the stack (Figure III-6). The emission of the sparklers is a safety hazard and was an indication of lack of thru compaction from the ram-briquetted materials. The materials had adequate energy content and could be handled by the train operators, but this result was unsatisfactory. The heat transfer to the boiler tubes was very good during the test, and the materials provided adequate energy to generate the steam required to run the engine. The flame characteristics are illustrated in Figure III-7. The briquettes also provided good hearth coverage.

In the second tests, torrefied pellets were employed. A screen had to be placed on the hearth to keep the pellets from falling through the grate. These materials provided good energy for the boilers, but their use was not deemed to be practical as materials closer in size to that of the coal typically used was desired.



Figure III-6: Sparklers emitted during full drafting of the boiler.



Figure III-7: Flame characteristics from ram briquettes.

Based on the results from the first trials at the Zoo, it became evident that more thorough compaction was a key need to have a usable fuel product. This became the focus of the development and rotary briquetting was thought to be the best option for achieving all the targets desired by Zoo personnel. In addition, based on our development experience, it was thought that in addition to total replacement of the coal with a pretreated biomass, partial replacement of the coal with a coal/torrefied fuel blend was desirable. Fuels were subsequently prepared—utilizing the full-densification system shown in Figure II-2—using both a blend of

coal and torrefied fuel and torrefied fuel alone. Enough material of both types was prepared to allow a full day of testing at the Zoo. Various characteristics of the produced fuels were measured after production: fuel content, drop durability, Kansas State tumbling can, crush strength, moisture uptake, grindability, and volatile matter/fixed carbon.

As can be seen in Table III-, the coal blend had a higher net caloric value compared to the 100% torrefied biomass material but also had significantly higher ash and sulfur contents. The typical coals used at the Zoo had both higher ash and significantly higher sulfur levels than that of the briquettes supplied during this test program.

Table III-1: Analytical comparisons for the fuel products tested.

Item	Torrefied Biomass	Coal/Torrefied Blend
Moisture (%)	4.91	4.73
Ash (%)	1.54	3.34
Volatile Matter (%)	61.53	64.66
Fixed Carbon (%)	33.68	27.27
Sulfur (%)	0.009	0.151
Net Caloric Value (kJ/kg)	20,763	23,166
Carbon (%)	58.22	60.76
Hydrogen (%)	5.28	4.71
Nitrogen (%)	0.27	0.53
Oxygen (%)	34.60	25.78

The physical properties of the two types of fuels provided were excellent and met most of the parameters highlighted in Section II of this report. The materials could be handled just like the coal typically used and were not dusty in handling or feeding to the boiler.

Both fuels were used during the day and provided adequate energy to allow the Zoo to pull a full-size train around the oval for multiple laps without difficulty. In addition, when the train was under full load and was under high draft conditions, the sparkler issue encountered with the use of the ram briquettes was eliminated. The locomotives were typically pulling a load of cars that approached 23,000 kg (~50,000 lb). The energy content of both material types was deemed adequate for use at the Zoo.

The other attributes of concern included comparisons with opacity and sulfur-related emissions as well as heat transfer to the boiler tubes. In addition, hearth coverage and hearth protection were also important concerns. Figure III-8 through Figure III-11 illustrate some of the key comparisons with coal.



Figure III-8: Hearth coverage under low-draft conditions.



Figure III-9: Combustion of fuel briquettes under full draft conditions.

Both the blend with coal and the 100% biomass blends worked well under low and high draft conditions, and as compared in Figure III-10, the two materials outperformed the coal in terms of more effective flame characteristics that would then enhance steam production for the boiler itself.

**Coal****Biocoal****Figure III-10: Comparison of flame characteristics for coal and torrefied biomass fuels.**

The stack emissions generated in using the fuels were noticeably improved when using either the 100% torrefied fuel briquettes or in using the blend, compared to the conventional coal employed. This is shown in Figure III-11 and Figure III-12.

**Coal****Biocoal Blend****Figure III-11: Comparison of stack emissions in using coal or coal/torrefied fuel blend.**



Figure III-12: Stack emissions for 100% torrefied biomass briquettes (steam release also shown at right of stack.)

After the full day of testing was completed, a discussion on the utility of using the new fuel products was undertaken with all involved in the test program, and the basic conclusion was that the new fuel briquettes were a good substitute for the coal they typically would use. The briquettes provided adequate power to pull the train, the emissions were greatly reduced, and the sparkler issue that had occurred with the ram briquettes was overcome using the rotary briquetting configuration employed to produce the fuel products.

Conclusions

- Mechanically sound briquettes from well-torrefied wood can be produced with high-energy density and provide adequate energy for the stationary hearth investigated.
- Torrefied materials can outperform industrial coal under rigorous firing conditions.
- Excellent-quality briquettes for commercial production were produced using one pocketed roll, one smooth roll.
- Raw starting material influences physical properties of final product and the type of compaction is a significant variable.
- Satisfactory products can be made at low binder levels, if a binder is required
- Final products exceed sub-bituminous coal properties and give excellent improvements from both a heat transfer and emissions perspective

Future Work

Much has been learned during this investigation. Significant optimization work can still be undertaken for the densification and properties development area. For example, the use of a commercial briquetting device provides significantly more thorough compaction than can be achieved under bench-scale conditions. The full degree of improvement in making densified fuel products that meet all the needs of potential end-users must still be pursued. The system needs for using torrefied fuels includes all aspects of logistics to a customer, storage of the material at the customer location, and the ability of the materials to be used without significant

equipment modification at the client facility. The demonstration facility now in place at our Institute can be a unique asset to the industry in making sure that any future production facility can meet the stringent needs of their potential clients through verification at the demonstration level, and this can be accomplished at the Coleraine, MN facility.

Acknowledgement

Financial support for this work was provided by the Consortium for Advanced Wood to Energy Solutions (CAWES), a jointly funded program by the US Forrest Service and the US Foundation for Forestry and Communities. The Coalition for Sustainable Rail and the Milwaukee County Zoo were instrumental in allowing the fuels testing using their steam engines and use of the park rail system. K.R. Komarek greatly assisted with supply and optimization of the B220 briquetting machine. The operating staff of NRRRI's Coleraine Renewable Energy Facility, in conjunction with our engineers and scientists, developed the necessary conditions to successfully produce high-quality fuels that could be densified to produce outstanding fuel products.

SECTION IV

Overcoming the Densification Challenges of Torrefied and Hydrothermally Carbonized Biomass: Key Learnings and Challenges

Executive Summary

The Natural Resources Research Institute has been engaged in work to develop demonstration level production of solid biofuel densified products that can be stored outside, have high bulk densities for ease of logistical transport, have good handling characteristics that minimize dust generation, grindability that is similar to coal used in power plants, and have fuel contents that match or exceed sub-bituminous coal levels. This work has been on-going since 2010. Over the course of this work, our team has extracted significant knowledge over the years and this document presents some of the unique challenges that our development teams have overcome.

During the work at the Institute, two pretreatment technologies have been investigated for concentrating the energy content of raw biomass. These include: torrefaction using an indirectly fired rotary kiln process at the demonstration level and hydrothermal carbonization (HTC) at the pilot scale. Early in 2011, the Sylva Corporation, Princeton, MN and the NRRI successfully operated a rotary torrefaction kiln and preliminarily established some of the key operating parameters of a rotary kiln system, including temperature profiles, particle size effects, throughput rates and a limited characterization of the torrefied fuel exiting the kiln. These early experiences demonstrated the operability of the rotary kiln system, established the steady-state atmospheric flare of the torrefied wood gasses and generated enough viable material for some initial grindability and pelletizing trials. This document highlights some of the key findings, challenges and opportunities our team uncovered during these early and subsequent trials.

Following these early challenges, the NRRI invested heavily in a Heyl and Patterson torrefaction rotary kiln system equipped with a rotary dryer and integrated with a variety of densification systems including rotary briquetting, ring and die pelleting and reciprocation ram compaction. The NRRI has found that rotary briquetting appears to be the most robust densification technique across the widest variety of material types. Although ram compaction and ring and die pelletizing work very well with hydrothermally carbonized (HTC) materials, the torrefied materials have been exhaustively difficult to densify unless they are blended with the appropriate amount of HTC materials or with non-torrefied or white wood.

The Institute has also collaborated with Syngas Technologies, Elk River, MN on a pilot scale vertical moving bed, directly heated steam-based process at the pilot scale and next year will install this technology at the demonstration scale. A key factor in showing the full technical feasibility of using the pretreated materials is to demonstrate that the produced particulate fuel products can be densified to a level that allows good logistical and handling practices to be routinely attained. It has been found that hydrothermally processed materials can be agglomerated using a variety of densification devices including pelleting and briquetting in a repeatable and practical manner using commercial densification equipment with and without the use of binders.

Torrefied materials have proven to be much more difficult to densify using various densification equipment especially as the degree of torrefaction increases. Uniformly torrefied

materials at high energy level appear to be especially difficult to densify but possess the attributes of high fuel value and good grindability with very little residual fiber content compared to less torrefied material or steam exploded biomass. The work undertaken and explained in the following has shown that highly torrefied materials can be satisfactorily densified to produce high energy content products that have good physical properties, possess acceptable moisture resistance, low ash, sulfur and mercury content, and have bulk densities that can lead to improved logistics. The densification practices involve optimizing overall process conditions on an integrated systems basis and include moisture level, densification pressure, mix preparation pressure, and the use of appropriate binders when required. The densification system that seems to show great promise for the highly torrefied materials is briquetting. Work will continue in examining other densification options and in improving the conditions used and reported in this report.

Evolution of Development at NRRRI

The Institute has worked on various levels to better understand both torrefaction and hydrothermal carbonization. Figure IV- and Figure IV-2 illustrate this development effort over time.

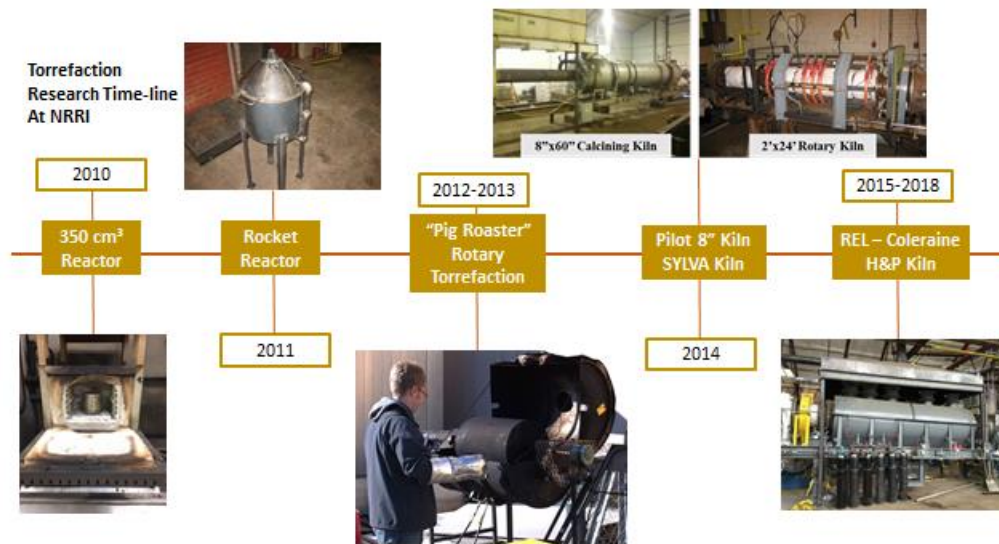


Figure IV-1: Evolution of torrefaction development over time.



Figure IV-2: Evolution of hydrothermal carbonization research.

Lessons Learned

Process and Equipment Limitations Identified

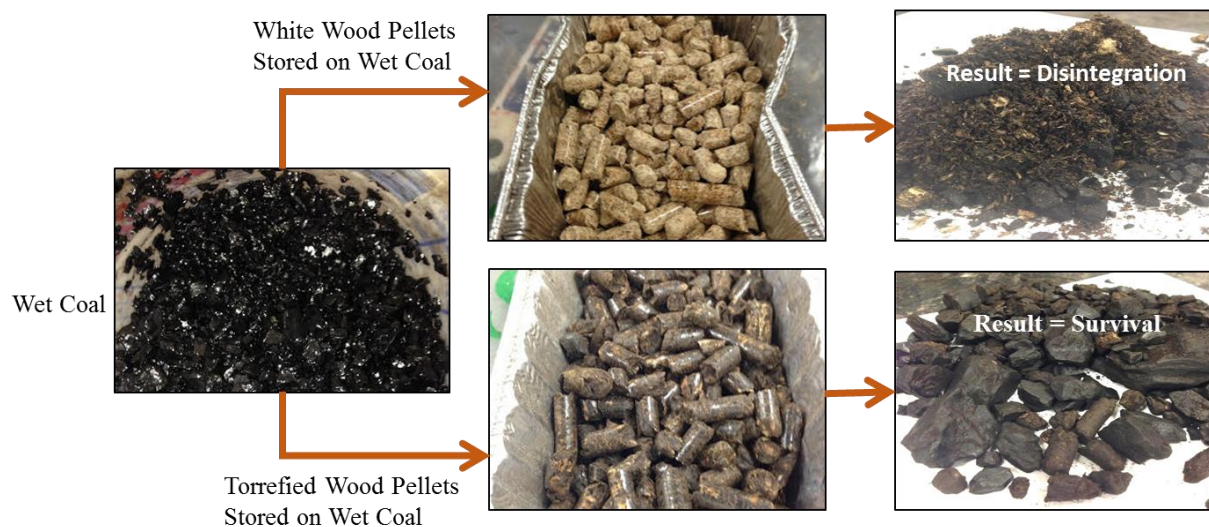
One of the equipment limitations our team identified early on concerned the grindability of the material. If the degree of torrefaction of the material is too low or the material is not uniformly torrefied, the tenacity of the under-torrefied fibers leads to difficulties in grinding. The fibrous material does not fracture easily. We found that the use of a turbulizer equipped with the appropriate impacting surfaces could accomplish both mixing and grinding in a single-unit operation simultaneously if well torrefied materials are produced.

Figure IV-3 demonstrates the variability when grinding different levels of torrefied materials with a turbulizer. Under torrefied material is too tenacious and results in poor grindability as evidenced by the lighter particles pointed out above and the resulting smaller portion of fines generated across the turbulizer. Although the turbulizer is not really designed to be a grinder, our team found that with an acceptable torrefaction level, the size distribution of material exiting the turbulizer was adequate for forming durable and moisture-resistant briquettes.



Figure IV-3: Torrefaction levels and the effect on grindability.**Outside Storage Limitations**

When co-firing pellets with wet coal, white wood pellets have limitations compared to torrefied wood pellets. White wood pellets degrade and disintegrate when they come in contact with wet coal particles. Conversely, torrefied pellets, if they are stored or come in contact with wet coal particles, survive and do not disintegrate readily. These attributes are clearly displayed in Figure IV-4, where white wood pellets, after being placed onto a simulated bed of wet coal particles, wick up the moisture, expand, and disintegrate back to their original state. Torrefied pellets survive and can be seamlessly handled in conventional coal handling systems.

**Figure IV-4:** White wood pellets versus torrefied wood pellets stored on wet coal bed.

Compaction Protocol Limitations

Compaction protocols must be developed for each specific material across several variables including mixing time, temperature, moisture, particle size, and retention time at specified pressure. One of the limitations our team identified when using an axial type Carver press was the “time-at-pressure” parameter. In general, for most materials, a 15-second retention “time-at-pressure” is required to get durable moisture-resistant briquettes. This makes scale-up to commercial equipment challenging, where “time-at-pressure” parameters are of the order of 1–2 seconds. Generally, the Carver press (Figure IV-5) is useful in determination of the minimum pressure to generate a durable moisture-resistant briquette at targeted particle size, moisture content, and binder type taken at a minimum 15-second retention time at pressure in the die. Then, as commercial runs are initiated, fine tuning may be required where more binder or more heating time prior to compaction is required.

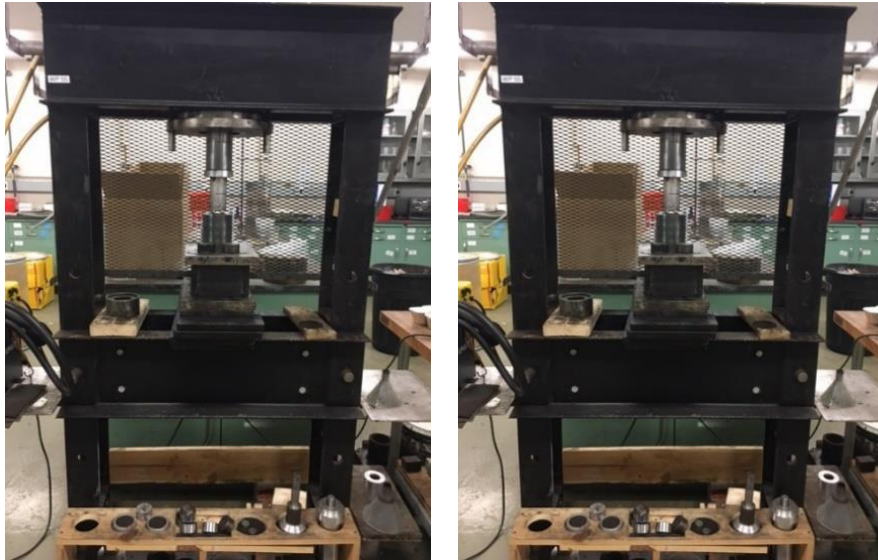


Figure IV-5: Carver press.

The limitation of “time-at-pressure” for briquettes formed at 2, 5, 15, 30, and 60 seconds is shown graphically in (Figure IV-6). As the figure shows, the briquettes really do not begin to hold their shape until after 15 seconds.



Figure IV-6: Limitations of “time-at-pressure” for Carver press scale-up.

For most torrefied materials, the ideal conditions for forming a durable moisture-resistant briquette are a pre-heat temperature 88°C – 104°C (190°F – 220°), a grind spec of 70% passing 30 mesh (0.595 mm), and a retention time of about 15–30 seconds. The ideal moisture content

is approximately 4–12% when loading the feedstock into die cylinder. However, there can be significant differences noted across different material types. For example, our team has found that with red oak, 4 % moisture content, and 70% passing 30 mesh (0.595 mm), and a temperature of 88°C (190°F) makes durable moisture-resistant briquettes on a commercial briquetter (Komarek B220B). However, to achieve the same degree of moisture resistance with red pine, a much higher temperature in the 99°C – 104°C (210°F – 220°F) range is required.

Influence of Briquette Geometry

One of the limitations identified for briquette geometry when using the Carver press was that with a radiused concave die, a density gradient is evident across the depth of the briquette (Figure IV-7a). Since compaction is happening in a single, downward motion, a natural gradient forms, where material with greater density is at the bottom portion of the die compared to the top portion. This characteristic would surface when testing durability, where the top portion of the briquette would abrade more easily than the bottom portion. To help ameliorate this effect, raised buttons were affixed to the top and bottom portions of the concave dies (Figure IV-7b). This helped to eliminate the gradient across the die and helped to improve durability significantly. This not only eliminates areas of lower density (on the spherical surfaces) but also creates air space within a pile of briquettes, thereby enhancing uniform combustibility.

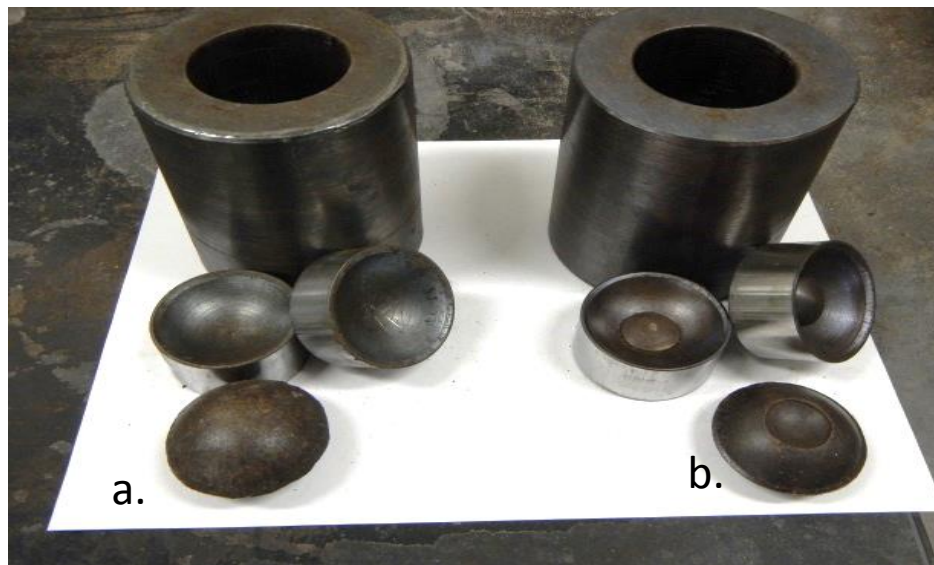


Figure IV-7: Concave die (a) and button die (b) effect on briquette density gradient.

Hydraulic Ram Briquetting

The B-100 hydraulic ram compactor (**Error! Reference source not found.**) was used to determine densification parameters of a variety of torrefied biomass feedstock with the use of a PVA binder system. Steam-exploded biomass as well as highly to moderately torrefied feedstocks were used. The operability parameters were determined, as well as the limitations of the B-100 (Table IV-1). Parameters identified included: hydraulic pressure, system operating clamp pressure, and feed rate needed to form viable briquettes. Grind specification was also evaluated; torrefied feedstock was hammer milled to a -9.525 mm (-3/8 inch) size specification. The take-away learnings from these initial compaction trials was that the -9.525 mm (-3/8 inch) grind specification, combined with axial compaction, were inadequate when actual briquettes were burned in a one-quarter-scale steam locomotive engine representing a stationary hearth condition. These cylindrical fuel briquettes were found to expand significantly when burned in an updraft, fixed-bed boiler. This resulted in significant “sparklers” emitting from the stack (Figure IV-9) of the steam locomotive. As a result, the team abandoned this compaction scenario when end use was in updraft, fixed-bed boiler systems.



Figure IV-8: B-100 ram briquetter.

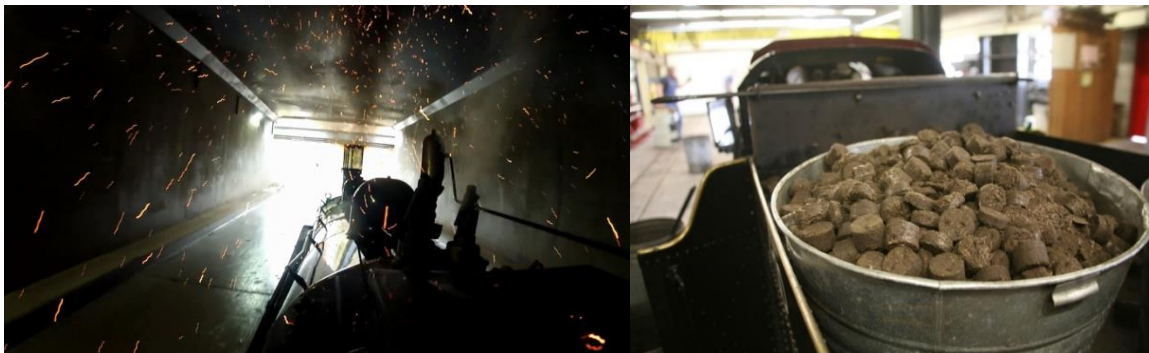


Figure IV-9: Sparklers from stack and finished briquettes.

Table IV-1: Operability parameters used in the commissioning trial runs on B-100 briquetter.

	Operating Clamp	Clamp setting		System
	Pressure	low high	Feed rate setting	Operating
Run	psig	psig	(Unitless)	Pressure, psig
1	800	1700/2300		0.4 2000-2300
2	800	1700/2300		0.4 2000-2300
3	1000	1700/2300		0.4 2000-2300
4	1000	1700/2300		0.4 2000-2300
5	1000	1700/2300		0.4 2000-2300
6	1000	1700/2300		0.4 2000-2300
7	500	1700/2300		0.4 2000-2300



Preliminary Binder Evaluations

Preliminary scoping trials were conducted across a variety of binder types, with durability and moisture-resistance (MPI) factors as metrics (**Table IV-2**). These scoping trials were undertaken to identify those binder systems that yielded both durability and moisture resistance. A moisture-penetration index was also used to eliminate those binders that would imbibe a droplet of water in less than 3,600 seconds. Those binder types that yielded MPIs of > 3600 seconds were selected as candidates for future investigation and scale-up. These initial scoping trials indicated that guar gum, wax, HTC birch bark, HTC wood, HTC switchgrass and steam-exploded wood could impart moisture resistance if included in a formulary and prepared properly.

Table IV-2: Preliminary Scoping Trials Characterizing Various Binders

Binder	Durability Factor	Moisture Resistance factor	Notes
None	Excellent when formed at high temperature and grind spec. of < 20 mesh (-0.841 mm)	Very good at 177°C (350°F) forming temperature and grind spec of < 20 mesh (-0.841 mm)	Hot forming at 177°C (350°F) with normal BTU achieves moisture resistance, not so good with high-BTU material ³ MPI > 3600 s
Calcium lignosulfonate	Good	Poor	MPI < 120 s
Sodium lignosulfonate	Good	Poor	MPI < 120 s
CaO	Good	Poor	MPI < 120 s
Ca(OH) ₂	Good ³	Poor	MPI < 120 s
CaCO ₃	Good	Poor	MPI < 120 s
Molasses	Good	Poor	MPI < 120 s
CaO/molasses	Good	Poor	MPI < 120 s
Xantham gum	Good ¹	Poor	Good binder for high-BTU material
Guar gum	Good ²	Poor	Good binder for high-BTU material
Bentonite	Good	Poor	MPI < 120 s
Asphalt tar	Good	Excellent	MPI > 18,000 s
Paraffin wax	Good	Excellent	MPI > 18,000 s
Hydrothermally modified birch bark tar	Good	Excellent	MPI > 18,000 s
Hydrothermally modified wood	Good	Excellent	MPI > 18,000 s
Hydrothermally modified switch grass	Good	Excellent	MPI > 18,000 s
Steam exploded wood	Good	Excellent	MPI > 18,000 s
Cellulose acetate	Good	Poor	MPI < 120 s
Brewex	Good	Poor	MPI < 120 s
Sodium silicate	Good	Poor	MPI < 120 s
Recycled tear-off shingles (TOS)	Good	Poor	Could be better if silica is removed

^{1,2}Good at low dosage, ³high btu material still has poor MPI even with high temp.

Influence of Tar and Wax Components on Ash Fusion Properties and Hardgrove Grindability Index (HGI)

One of the concerns with some of the binder systems was their potential negative influence on ash fusion properties, HGI, and sulfur content. A series of experiments were conducted at 10% binder inclusion with torrefied wood and the sulfur, heating value (HHV) and ash fusion temps measured (Table IV-3 and Table IV-4).

Table IV-3: Characterizations of tar and wax binder variations and the control.

Binder ¹	HGI	HHV (btu/lb)	Sulfur (%)	Ash (%)	Ash Fusion (oxd)	Ash Fusion (red)
Control	30	9,176	0.06	1.89	>1449°C	>1449°C
Crafco Tar (fossil)	27	9,515	0.261	3.46	>1449°C	>1449°C
B.B.Tar (natural)	38	9,615	0.12	1.62	>1449°C	>1449°C
5828 Tar (fossil)	19	9,785	0.475	2.67	>1449°C	>1449°C
Paraffin Wax (fossil)	13	10,174	0.042	2.34	>1449°C	>1449°C

¹All binders tested at 10% dosage formed under similar conditions (**1 BTU=1.055 kJ**).

Table IV-4: HGI ratings.

HGI Reading	Difficulty Level
<40	Very Hard
40-60	Hard
60-80	Moderately Hard
80-100	Soft
100-120	Very Soft
>120	Extremely Soft

All the binders generally showed an improvement in heating value, and surprisingly, none of them lowered the ash fusion temperature compared to a control. There was no notable trend evident in the HGI data, other than to note the particles were found to stick to the grinding balls, and this likely influenced the HGI test results. **It was concluded that the HGI was not an appropriate test for evaluating binders due to the limitations of binder particles adhering to the grinding balls, and this merits more consideration when choosing test materials.** Paraffin wax, for example, may stick onto the grinding balls, resulting in difficulty in grinding and material recovery. This can lead to erroneous measurements of this index. Craftco tar and P5828 tar are asphalt tars, resulting in higher sulfur content than the others listed. The increased ash content of Crafco tar, P5828 tar, and paraffin wax is likely because they were formed from a fossil base material. Our conclusions are that HGI should only be used for well-torrefied and uniformly torrefied materials without any binder. The influence of binder sticking to the ball charge, combined with under-torrefied or non-uniformly torrefied material, only makes the HGI test yield erroneous harder-to-grind values. The validity of any HGI test should be highly scrutinized whenever it is used on torrefied materials.

Influence of Pressure and Binder Dosage with Button Face vs Concave Die at 3 Second Retention Time.

The Oriox 255/PVA binder system was evaluated at 2% and 0.7% dosage levels with a button face and concave die (**Table IV-5**; Figure IV-10). Our findings revealed better durability was achieved with the button-faced die, and binder dosage could be reduced to the 0.7% level. In addition, the incorporation of the PVA allowed retention times to be reduced to the three-second range. All of the tests in **Table IV-5** were done at three-second die retention.

Table IV-5: Durability performance of Oriox 255/PVA Binder with concave versus button die.

2% Oriox 255/PVA Binder with Concave two-inch die	
Pressure (Bars)	Durability, % Survival
240	53.3
479	96.8
958	98.4
1438	99.4
2% Oriox 255/PVA Binder with Button two-inch die	
Pressure (Bars)	Durability, % Survival
240	63.7
479	98.8
958	99.4
1438	98.0
0.7 % Oriox 255/PVA Binder with Button two-inch die	
Pressure (Bars)	Durability, % Survival
958	98.1
958	99.5

*All samples used three-second die retention time, 958 bar, and microwave heat to 10% moisture to 88°C – 99°C (190°F – 210°F).



Figure IV-10: Concave briquette versus button briquette.

Strategies to achieve Moisture Resistance with Steam Explosion

The objective for this experiment was to determine the use of a steam explosion feedstock as a binder for torrefied wood to impart moisture resistance. We received steam-exploded loblolly pine from Herty's steam-explosion lab. The steam-explosion conditions for the two samples received were: 165°C, 30 min, Severity Factor = 3.4(B), and 175°C, 90 min, Severity Factor = 4.2(D). Briquettes consisted of various levels of steam-exploded material as binder ranging from of 100% steam-exploded material and a combination of 10, 20, and 30% steam-exploded to torrefied materials. The briquettes were 50.8 mm (2 inch) diameter (concave die) and made individually on a 50-ton H Frame press at 1,000 bar and under microwave preheating conditions of 88°C – 99°C (190°F -210°F). The briquettes were then placed on a simulated wet coal bed to observe any moisture uptake or disintegration (Figure IV-11). **What was learned from this experiment was that steam explosion at 165°C – 175°C was not enough to impart moisture resistance, as virtually all of the briquetted materials wicked up moisture from the simulated coal bed, expanded significantly, and opened any bonds that were formed during the compaction process.** This was also learned during the commissioning of the hydraulic ram B-100, where steam-exploded feedstock was used as a binder for torrefied wood briquettes. These briquettes, when exposed to moisture, would also disintegrate and break apart.



100% SE binder B before and after wet coal exposure. Moderate disintegration noted after 3.81% moisture uptake.



100% SE binder D before and after wet coal exposure. Severe disintegration noted after 8.24% moisture uptake.



30% SE binder B/70% T-Wood before and after wet coal exposure. Severe disintegration noted after 9.81% moisture uptake.



30% SE binder D/70% T-Wood before and after wet coal exposure. Severe disintegration noted after 6.86% moisture uptake.



10% SE binder B/90% T-Wood before and after wet coal exposure. Severe disintegration noted after 6.93% moisture uptake.



20% SE binder D/80% T-Wood before and after wet coal exposure. Severe disintegration noted after 9.34% moisture uptake.

Figure IV-11: Steam explosion binders B or D, and torrefied wood formulations and moisture resistance characterization.

Coating Strategies for Imparting Moisture Resistance

Various samples of binders, briquette formulations, and coatings were tested to determine if any were feasible to impart moisture resistance. The description and results are noted in Figure IV-12.



A thin coating of hot wax can be spray coated onto the briquette surface. But the coating is easily sloughed off upon extensive handling.



Hot forming the briquettes at 200°C (392°F) imparts significant moisture resistance--- but this temperature range is outside the capabilities of commercial compaction equipment.



Application of a powder wax followed by heating can imbibe wax into the surfaces of the briquette. But upon weathering the coating may slough off.



Double compaction agglomeration: anticipated lower surface area with the addition of HTC binder and moisture resistant individual particles. Instead, pre compacted particles resulted in uneven mating surfaces providing a conduit for moisture penetration between particles.



Course ground particles versus fine ground particles offered no difference in moisture resistance when white wood pellets were torrefied, crumbled, and then re-agglomerated.



When white wood pellets are torrefied and then crumbled and sized, they offer excellent moisture resistance and low dust content. These crumbled and sized agglomerates could easily be stored outside. But bulk density is relatively low at 512 kg/m³ (32 lb/ft³).

Figure IV-12: Coating and densification strategies to achieve moisture resistance.

Hydrothermal Carbonization – Influence of Time, Temperature, and Grind Specification across Birch, Pine, and Gum Tree from Australia

The influence of time, across a temperature range of 220°C – 235°C and across various material types and grinds showed that the hydrothermal process has resulting energy contents in the 25,500 kJ/kg (11,000 BTU/lb) range (**Table IV-6**; Figure IV-13). In addition, all of the resulting materials are easily compactable with any densification device including ram compaction, rotary compaction, and ring and die pelletizing. This shows the robustness of the HTC conversion regardless of wood type, and, whether it is chips or ground through a hammermill, the resulting material has significant coal-like properties.

Table IV-6: Various HTC characterizations.

Material	Temperature (°C)	Time (min)	Energy Content (kJ/kg)	Sulfur Content (%)	Ash Content (%)
Whole tree Birch Chips	220	180	25,656	0.035	0.58
Birch Bark Waste – Unground	226	60	25,772	0.04	1.82
Birch Bark Waste – Ground	235	15	26,993	0.034	1.93
Birch Bark Waste – Unground	235	15	26,823	0.034	1.91
Pine – Whole Tree Chips	235	15	25,886	0.015	0.54
Gum Tree – whole Chips	235	15	25,491	0.014	0.21



Figure IV-13: Raw material to energy mud – the hydrothermal carbonization process.

Moldability and Release Character of HTC

One of the unique features of HTC materials is that it is very moldable and easily releases from complex die shapes (Figure IV-14).



Figure IV-14: HTC waffle briquettes.

Binding Properties and Moisture Resistance of Steam Stripped Torrefied Feedstock

An important finding that our team continues to uncover is the importance of the surface preparation. If super-heated steam is used as a sweep gas, our team hypothesizes that the surface character of the material may become *more amenable to bonding as condensable volatile matter is removed from the surface of the torrefied material.*

The moisture-resistant properties of steam-stripped torrefied briquettes made from either black ash or tamarack were compared to 100% HTC briquettes (Figure IV-15). Black ash had the most moisture resistance (lowest moisture uptake) among all the samples including the HTC briquettes. The steam-stripped torrefied briquettes were formed using hot dies, whereas the HTC briquettes were formed using room-temperature dies. This is a variable to consider when comparing the two sample classes. This experiment outlined the possibility that *steam stripping when torrefying may prepare the feedstock to a level that may be comparable to HTC.* The NRRI continues to bolster the case for steam stripping.

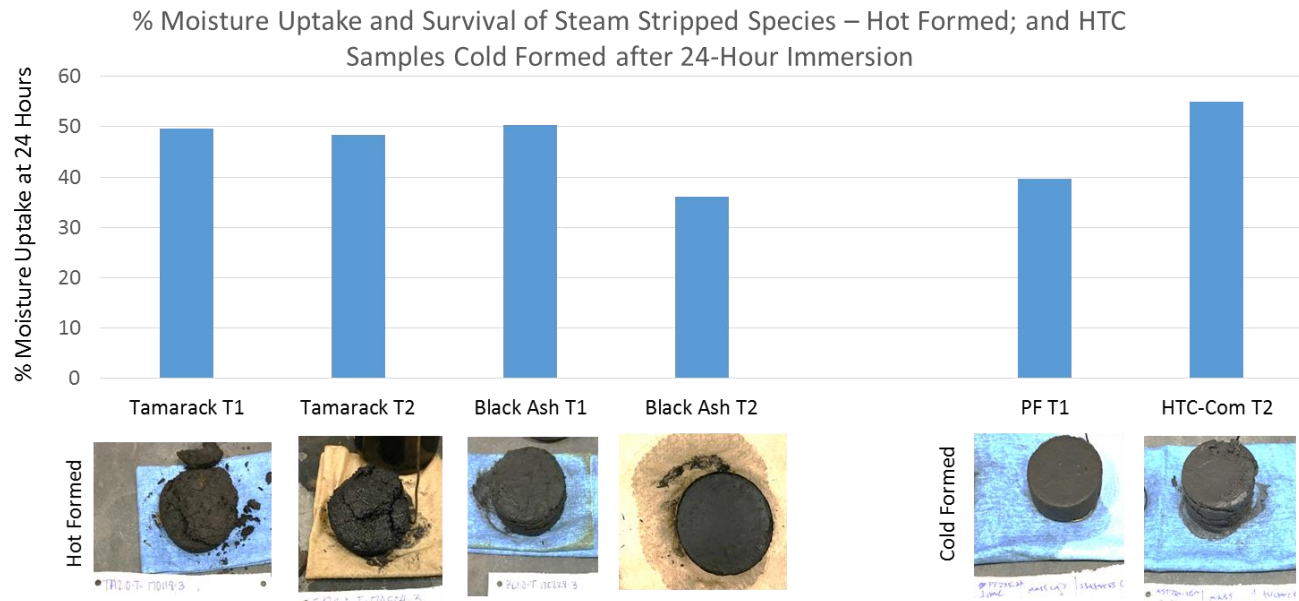


Figure IV-15: Moisture uptake of steam-stripped briquettes versus HTC briquettes.

We believe that the surfaces of steam-stripped wood may provide a cleaner surface for significant bonding. In an effort to show this microscopically, our team torrefied wood chips with and without the use of a steam-stripping stage. We then took photographs of the steam stripped vs non-steam-stripped materials. Indeed, the steam-stripped surfaces appear to be less blackened, indicating that the steam may act to cleanse the surface of condensed volatile matter and thus provide a much cleaner surface for bonding to occur. This hypothesis was also substantiated by collecting the blackened drippings emanating from a pilot rotary kiln. It was thought that these blackened liquid drippings, if they were effectively re-mixed with torrefied wood, could provide some additional bonding character to the base torrefied material. However, the opposite was true, and the observation was that the surface was compromised rather than improved. This led our team to believe that the surface character of the material, if it were cleansed with steam, may prevent re-deposition of condensable gases and provide better bonding character. Our team thinks that stripping off the VOCs may actually help to reduce the ignitability of the base fuel as well. We continue to actively pursue a rigorous confirmation of this hypothesis. As can be seen in the SEM analysis in **Error! Reference source not found.**, the surface of steam-stripped wood appears to be more aggressively altered than the non-steam-stripped wood, thus creating more surface area for bonding to occur.

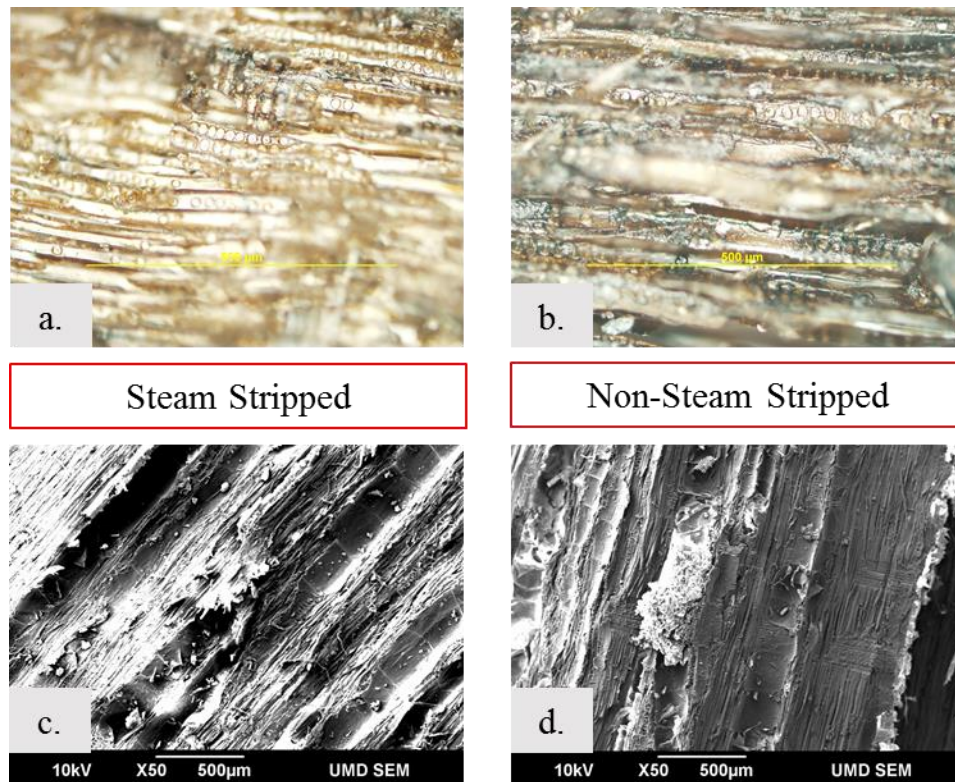


Figure IV-16: Microscopic and SEM analysis of steam-stripped torrefied wood versus commercially.

Moisture Resistance with Increasing Inclusions of HTC Binder

The two pictograms in Figure IV-17 display the level of disintegration over a 24-hour period of briquettes composed of pilot HTC (HTC-Com T2) and commercially torrefied wood at inclusion levels of 0 – 100%. The second set of briquettes was formulated with birch bark waste HTC (PF T1) and commercially torrefied wood. The briquettes have increasing percentages of HTC added. ***The learnings here include that the higher percentage of HTC added, the more moisture resistance is attained.*** Also learned from this experiment is that the temperature and retention times of the HTC preparation also influence moisture resistance. The pilot HTC (HTC-Com T2) was prepared at 220°C for 180 minutes, whereas birch bark waste HTC (PF T1) was prepared at 235°C for 20 minutes. Overall, the birch bark waste HTC briquettes imparted more moisture resistance at lower levels of inclusion than the commercial HTC briquettes.

The two pictograms in Figure IV-18 demonstrate the moisture resistance of briquettes composed of the two HTC species, as listed out in the previous paragraph, and combined with steam-stripped torrefied wood. Between the two sample sets in Figure IV-18, moisture resistance was attained with as little as 20% HTC inclusion; however, the briquettes formulated with pilot HTC had greater disintegration at 20% than those formulated with birch bark waste HTC. This points to the greater activity and functionality of the HTC formulated with the certain biomass materials.

24 Hour Immersion of Torrefied Wood

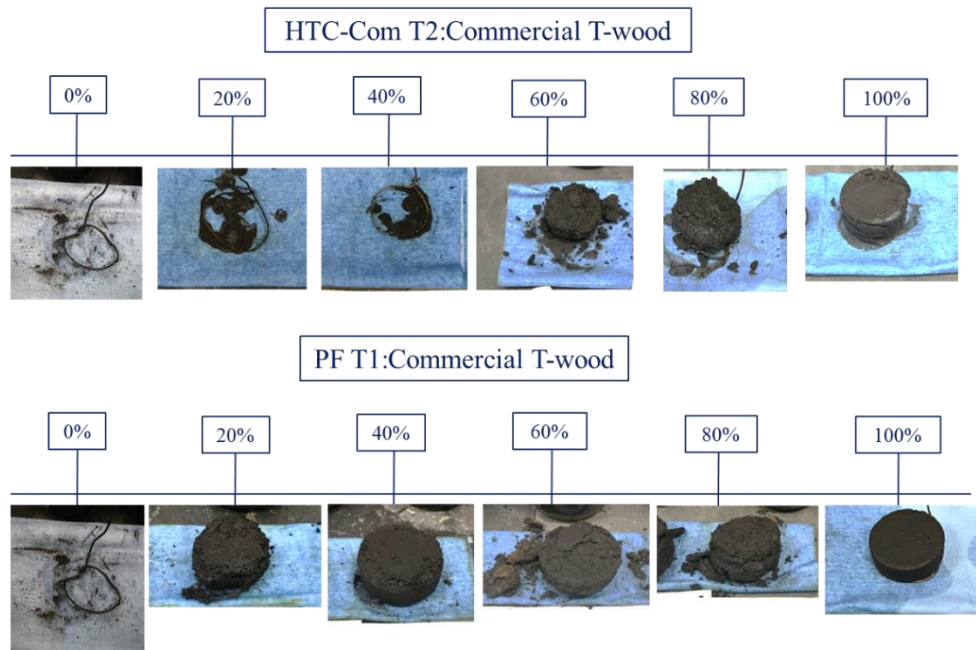


Figure IV-17: 24-Hour immersion of commercial HTC versus birch bark waste HTC briquettes formulated with commercially torrefied wood.

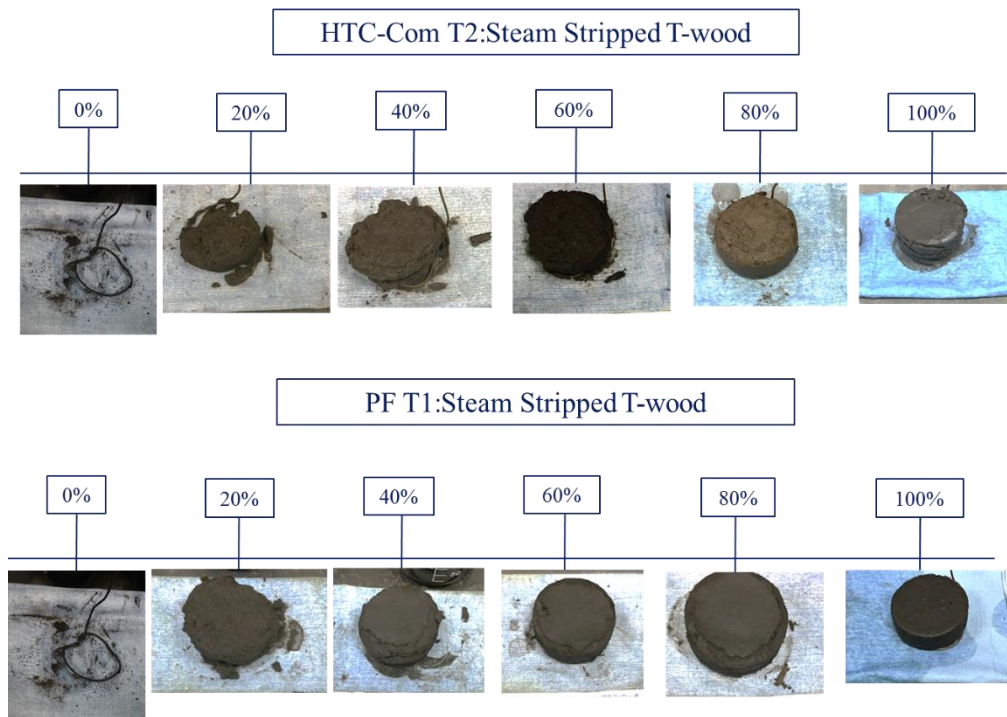


Figure IV-18: 24-Hour immersion of commercial HTC versus birch bark waste HTC briquettes formulated with steam-stripped torrefied wood.

The learnings from the four sample sets highlight the importance of the HTC preparation. ***A higher temperature and lower retention time seem to impart more moisture resistance at lower inclusion levels than the HTC formulated at lower temperatures and a longer retention time.*** Also garnered from this experiment is that steam stripping when torrefying versus traditional roasting can also impart moisture resistance. ***A clean surface preparation appears to be one of the drivers for achieving moisture resistance.*** Of the briquettes made using commercial HTC, those combined with steam-stripped torrefied feedstock had drastically more moisture resistance than those combined with commercially roasted torrefied wood.

Commercial Pellets Formulated with Commercial HTC Material

The NRRI contracted with a pilot HTC Facility to have approximately 500 kg of whole birch tree wood chips (bark-on) processed at 220°C for 180 minutes. This HTC material was then air dried and formed into pellet formulations at a commercial pellet facility. A commercial torrefied wood was used to blend with the HTC birch at ratios of 50% T-wood/50% HTC and 70% T-wood/30% HTC. All of the pellets produced at the commercial facility survived direct submersion in water for 24 hours (Figure IV-19). The specific die specifications that were used is proprietary.

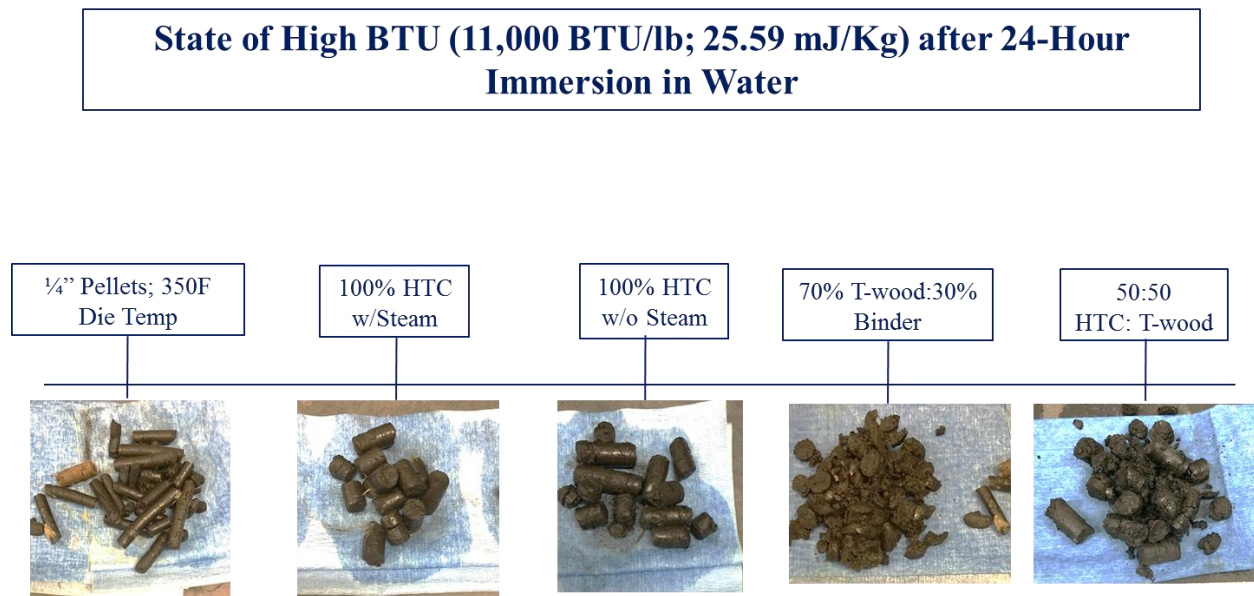


Figure IV-19: 24-hour immersion of high BTU/lb pellets.

Hydromulch for Outdoor Storage of Bulk Piles of Pellets or Briquettes

Scoping trials were investigated to explore the use of reactive hydromulch as a protective barrier to cover bulk piles of pellets for outdoor storage. Initial hydromulch blends incorporated 50% pulp mill residue with 50% refined wood fiber. A polysaccharide-type binder system was used. Small plates of pellets were covered with the reactive mulch formulation and allowed to harden. The hardened mulch layers were then wetted repeatedly with water droplets to see how they would shed moisture and disperse the droplets to not allow penetration into the pile. Inspection of the pellets in Figure IV-20 indicated minimal disintegration. This type of a protection strategy could be implemented in more tropical or humid climates and as an extra

measure to protect bulk piles from rain exposure and any subsequent degradation caused by this exposure. Various formulations can be developed by NRRI for this purpose.



Figure IV-20: State of pellets and briquettes after wetting hydromulch covering.

Scoping Trial for Use of Outer Birch Bark as Binder to Torrefied Wood

One of the favorable aspects of outer birch bark is that it is highly concentrated in triterpenes. Triterpenes are very hydrophobic, and thus would be a good choice as a starting material for achieving moisture resistance. Our initial trials revealed that even at inclusion levels of 10%, the HTC outer bark provided a high degree of moisture resistance. HTC outer birch bark material, in combination with conventional torrefied wood, all survived 24-hour immersion tests (Figure IV-21).

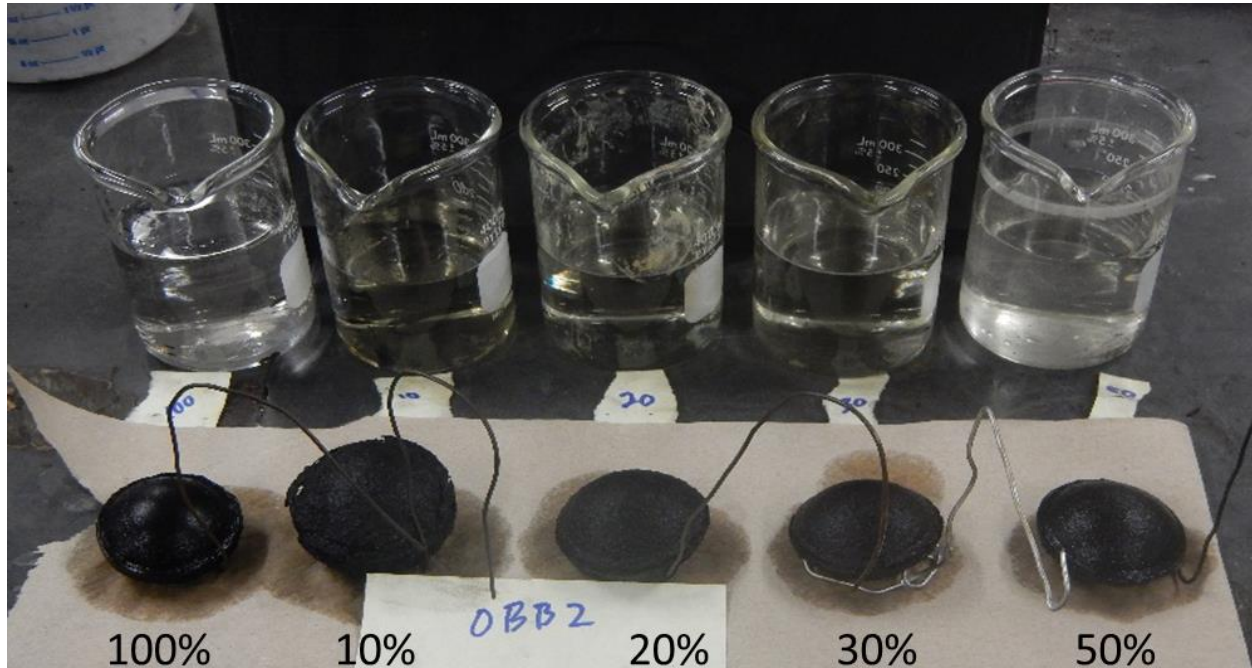


Figure IV-21: 24-hour immersion of outer birch bark binder and torrefied wood briquettes at various inclusions.

SYLVA Corporation Torrefied Kiln Runs, February 2011

In winter of 2011, the NRRI, in cooperation with the Sylva Corporation, conducted a series of torrefaction test runs using a modified indirectly heated rotary kiln. These tests were conducted to preliminarily validate the operability of a rotary kiln across a variety of wood chips sizes using mixed hardwoods and soft woods. The kiln was 0.61 m (24 inches) in diameter and approximately 7.3 m (24 ft) long and was heated indirectly with an annular center tube fired with fuel oil spanning its length. Over a period spanning several weeks, the Sylva/NRRI operating team prepared and air dried coarse and finer-sized wood chips to demonstrate the steady state operability of the rotary kiln system. The key findings in these early trials demonstrated the following:

1. The steady state capacity for the kiln was ranged from 31.8 kg/h – 113.6 kg/h (70 lb/h – 250 lb/h), depending on the dry solids loss across the kiln.
2. At a steady-state operating condition of 63.6 kg/h (140 lb/h) and at an average dry solids loss across the kiln of 28% to 30%, the three temperature zones in the reactor could be maintained at 310°C (590°F) in the front section, 316°C (600°F) in the center section, and 321°C (610°F) in the breach section of the reactor (Table IV-7).
3. Under the conditions stated above, the torrefaction gasses generated were successfully flared routinely to atmosphere (Figure IV-22).
4. The torrefied material exiting the kiln was misted with water spray and collected into 55-gallon drums. As the drums became full, lids were fastened securely and stored for subsequent fuel characterization.

5. One of the drums, after being stored over-night and outside, generated a negative pressure atmosphere within the drum and collapsed under the forces of vacuum generated.
6. Several super sacks of the torrefied material were brought to the NRRI for initial pelletizing and grindability trials. These early tests indicated that pelletizing with a typical ring and die pellet mill was quite challenging and difficult with conventional die specifications. Many die pluggings were encountered, with parallel frictional heat of material that caused temperature to rise into the plus 177°C (350°F) range, which is significantly outside the bounds of conventional pellet machines. However, the ease of grindability of the torrefied chips was improved several fold over the dry chip feed stock.
7. The uniformity of torrefaction across the kiln was heavily influenced by the particle distribution of the incoming feedstock. Larger chips required longer retention time than smaller chips to be uniformly torrefied throughout.
8. The proportion of finer material intermixed with the feedstock significantly influenced the operability parameters of the kiln. More fines resulted in significantly more operator intervention and fine tuning of feed rate, kiln speed, and water spray to directly cool the material.
9. With tightly sized wood chip feedstock spanning from 12.53 mm to 2.38 mm (½ inch x 8 mesh), the operability parameters required very minimal operator intervention. However, as the proportion of minus 2.38 mm (8 mesh) fines in the mix increased, the operability parameters were influenced significantly with more operator fine tuning and intervention were required.

Table IV-7: Typical calorific values for the torrefied wood operating with a kiln breach temperature of 321°C (610°F) over a sustained operating period of 14 hours at 63.6 kg/h (140 lb/h).*

Sample Number	Calorific value (dry basis), kJ/kg
1	27,481
2	24,498
3	25,800
4	22,265
5	24,087
Average	24,825

*Mixed hardwood/soft wood species, average dry solids loss ~28%.



Figure IV-22: Photographic detail of the modified Sylva Rotary Kiln showing tor gas flair to atmosphere and material flow collection into steel drum.

Co-firing Trial at a major 56,000 Tons/Year Pulverized Coal Utility

The NRRI/Sylva operating team also conducted a co-firing trial at a major pulverized coal-fired utility in August 2010. This trial was originally scheduled to fire torrefied wood chips and would have required truck load quantities to be delivered to the test site and ground in their coal pulverization system. However, because this volume was not available, an alternative dry sawdust powder was injected into the combustion system downstream of the pulverization system and just before entering the boiler. A positive displacement blower mounted on the end of a mulch delivery truck blew approximately 2,727 kg (6,000 lb) of dry sawdust into the combustion system at the utility. Select photographs of the mulch truck and the injection point into the combustion system are shown in Figure IV-23. Significant reduction in both mercury and sulfur dioxide, shown in the graphs (Figure IV-24 and Figure IV-25), were recorded throughout the steady state run.



Figure IV-23: System used at coal utility.

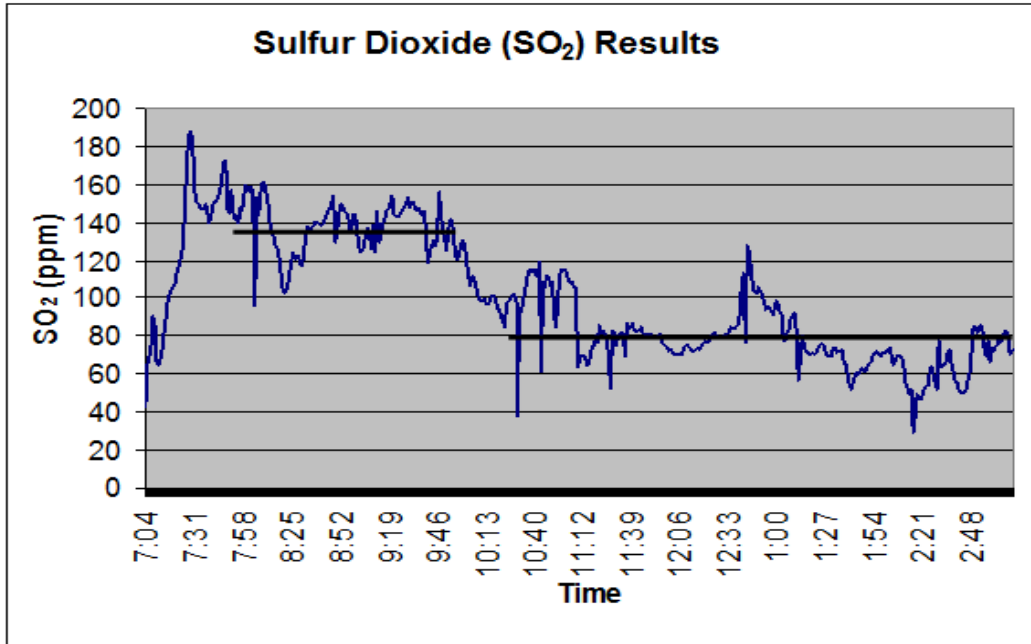


Figure IV-24: Sulfur reduction through use of biomass.

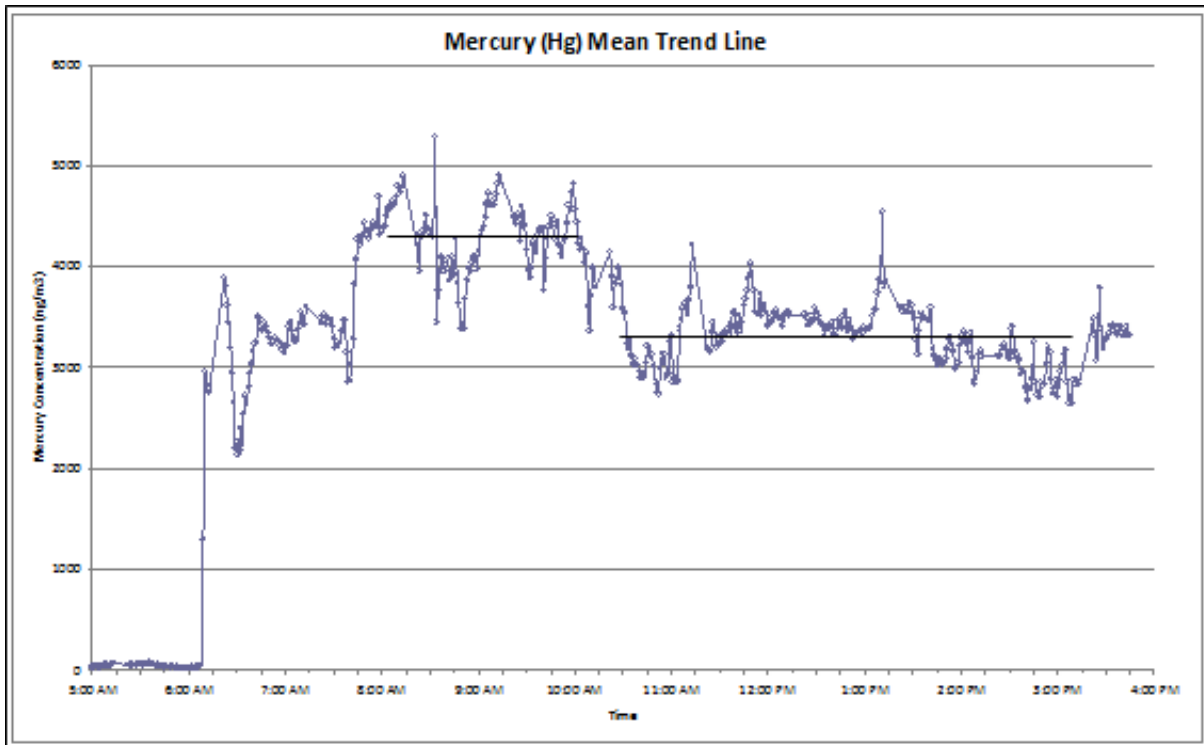


Figure IV-25: Mercury versus Time. Reduction of mercury utilizing biomass.

The following conclusions are drawn from this co-firing study:

1. Co-firing dry saw dust at nominal 682 kg/h (1,500 lb/h) into a conventional pulverized coal fired boiler is technically feasible.
2. A reduction in mercury concentration on the order of 23% was recorded throughout this trial. The reduction closely parallels the proportion of injected sawdust. The average coal-only and co-firing concentrations were 4,294 ng/m³ and 3,286 ng/m³, respectively.
3. The SO₂ concentration in the gas stream decreased significantly as a result of co-firing with sawdust. The average coal-only and co-firing concentrations were 137 ppm and 78 ppm, respectively.
4. Sparklers in the combustion zone appeared to be manageable and were not observed to reach the baghouse.

The results clearly demonstrate reductions in both mercury and SO₂ can be expected when co-firing dry sawdust into a conventional pulverized coal fired boiler. Co-firing appears to provide a logical pathway to meet evolving and more stringent future emission standards. A co-firing option may give utilities an additional tool to overcome the costs associated with expensive pollution control equipment. Similar results will occur in using advanced biofuels such as torrefied materials or other combination noted previously.

Advanced Biofuels can be produced in Various Agglomerated Shapes

NRRI has learned how to make a variety of shapes of consolidated products (Figure IV-26). The properties of the products and ease of densification require manipulation of assorted variables, depending on the equipment employed. ***An intimate knowledge of the raw materials to be used and the customer targets for the final products is required to efficiently produce desirable fuels.***



Figure IV-26: Various products can be made from the advanced biomass fuel products.