Development and Evaluation of Biochar-Filled Sheet Moulding Compound (SMC) for Low-Shrink and Fire-Retardant Formulations

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1. Abstract

Using forestry waste to create biochar is an appealing method of carbon sequestration to reduce greenhouse gas emissions. There has been research interest in the prospect of using biochar as an additive into both thermoplastic and thermoset polymers. However, no published research has been conducted where biochar is used as an additive in sheet molding compound (SMC), which is one of the most popular composite materials used in the automotive industry. This study is focused on investigating the impact of biochar fillers on SMC formulations. To investigate the impact of biochar fillers, two generic SMC formulations (Unsaturated Polyester Resin (UPR) matrix with 30%wt glass fiber) were selected, low-shrink (primarily filled with calcium carbonate (CaCO3)), and fire-retardant formulations (primarily filled with Alumina Trihydrate (ATH)). Control formulations of these generic SMC formulations were compared to a formulation where a portion of the standard filler (CaCO3 or ATH) from these generic formulations was replaced with biochar (40 PHR replaced with 20 PHR). Prior to compounding these SMC formulations, a viscosity study was conducted to understand the difference in resin demand between the industrially standard fillers and the biochar. After the viscosity study, formulations for the SMC were generated, and these materials were compounded using an SMC line. After a maturation period the materials were compression molded into parts using a 2500 ton compression molding press and an 18” by 18” square plaque tool. The parts were waterjet cut (ProtoMAX waterjet cutter) to extract test samples. The materials were mechanically (tensile, flexural, and impact) and thermally (TGA, and UL94) tested to investigate the impact of the biochar filler on the material properties of the SMC. The mechanical testing results showed that the biochar SMC had a lower density than the control formulations while maintaining similar mechanical properties. The thermal testing results showed that the biochar SMC was not as flame retardant as the industrially standard fire-retardant formulation.

2. Introduction

Canada has one of the worlds largest forestry industries which contributes more than $20 billion to the countries GDP [1]. Waste products from the forestry industry can contribute to green house gas emissions. Forestry waste can be pyrolyzed, and the carbon content of the forestry waste can be sequestered in a material known as biochar. Carbon sequestration is seen as a critical strategy to mitigate greenhouse gas emissions by Natural Resources Canada [2]. Biochar has already found an application in the improvement of soil quality [3], however, there is research interest into biochar filled polymers as another possible application for the material.

The majority of the research focus on the incorporation of biochar into polymers has been focused on incorporating the biochar as the sole filler in a polymer. This research addresses the question of how biochar fillers can impact the properties of polymers, however, it is limiting because more complex material formulations used for industrial products have not been investigated. Sheet molding compound (SMC) is one of the most popular composite materials in the automotive industry and has been established in the industry since the 1970's. Given the large volumes of automotive parts, and the long service life of automobiles, SMC materials would be a great candidate for a biochar filled composite material. Some applications of SMC materials in the automotive industry include: hoods, deck lids, fenders, radiator supports, bumper beams, roof frames, door frames, engine valve covers, timing chain covers, oil pans, exterior and interior body panels, trunk covers, lift gates, cargo boxes, radiator supports, and cross members [4].

In this study the impact of biochar fillers on SMC material performance were evaluated. This was achieved by compounding control batches of SMC (without biochar) and then biochar SMC (where a portion of the standard SMC fillers are replaced with biochar) and comparing the control material to the biochar SMC material. After the material is compounded the materials are compression molded and the compression molded parts are cut into test samples to perform the following testing: tensile, flexural, impact, TGA, and UL94. The results from the tests are compared and conclusions are drawn from this data.

3. Methods

This section covers the details of the creation of the parts for the experiment.

3.1. Materials

The materials used for the experiment as well as some information about their function and physical properties are listed in this section of the paper.

3.1.1. Matrix Materials

For the main resin component, unsaturated polyester resin was used. This resin is used for Class A and Lightweight Class A SMC applications for the automotive industry. To mitigate the shrinkage of the unsaturated polyester it is common in SMC formulations to include a low-profile additive (LPA) which will help reduce shrinkage and increase part surface quality, for this purpose an LPA was also used. To initiate the maturation, process a thickener is needed, in this experiment a standard MgO based thickener was used. To cross link the thermoset resin and initiator is needed, a common initiator for SMC production is tert-Butyl Peroxybenzoate (TBPB) which was used for this study. The basic properties of these materials are shown in the table below.

Table 1. Matrix material liquid component basic properties.

Material	Viscosity (cps)	Density (g/cm^3)
Unsaturated Polyester Resin	1800	1.09
Low-Profile Additive	250	1.02
Thickener	9500	1.38

3.1.2.Solid Additives and Filler Materials

The most common filler for SMC formulations is calcium carbonate. For the low-shrink SMC formulations calcium carbonate was used as the primary filler. For fire-retardant (FR) SMC formulations the most common additive is alumina trihydrate (ATH). ATH was used as the primary filler for the FR formulations. In SMC formulations it is common to use zinc stearate as an internal mold release, so for the formulations compounded in this study zinc stearate was used as an internal mold release. The calcium carbonate, ATH, and zinc stearate represent typical materials used for industrial SMC production. The purpose of this study is to investigate the impact of a non-traditional filler in SMC production which is biochar. The basic properties of these materials are shown in the table below.

Table 2. Solid additives component basic properties.

Material	Particle Shape	Particle Size (µm)	BET Surface Area (m^2/g)
Calcium Carbonate	Irregular Blocky	6.0	2.1
Alumina Trihydrate	Irregular Blocky	10.0	2.8
Hard Wood Maple Biochar	Irregular Pins/Shards	4.8	300
Zinc Stearate	Irregular Plates	6-10	N/A

Figure 1. Hardwood maple biochar.

3.1.3. Fiber Reinforcement Materials

For the large volumes of automotive industry and the typical structural requirements needed for the historical applications of automotive SMC glass fibers are most often the reinforcement of choice. The glass fibers used in this experiment were Johns Manville Multistar 264.

Table 3. Glass fiber basic properties.

Material	Filament Diameter (µm)	Linear Density (tex)	Density (g/cm^3)
E-Glass Fiber	14	4800	2.6

3.2. Drying Biochar Filler

It is desirable to maintain a low moisture content in the fillers added to SMC formulations. An ideal SMC filler would have a moisture content below 0.1% [5]. Moisture in the formulation can create several problems, the two main concerns is than moisture can impact the thickening behaviour of the SMC paste and too much moisture will result in part blisters after molding.

Biochar is known to be hydrophilic and readily absorb moisture from the atmosphere. The moisture of biochar was measured using a Mettler Toledo Moisture Analysis machine, natural moisture content when stored in lab conditions was 3.21%. Which is much higher than the ideal 0.1%wt moisture content. As a result, the biochar will need to undergo a drying process before it can be incorporated into the SMC paste. The biochar was placed in an aluminum foil container and placed in a circulating air oven set at 85 ˚C for 24 hours and the moisture content of the biochar was reduced to 0.82% which is still much higher than the ideal moisture content, however not as much biochar filler will be used in the paste formulations so it was deemed to be an acceptable moisture content.

3.3. Viscosity Experiments

3.3.1. Basic Filler/Resin Combinations

In this section the viscosity measurements are all gathered from the combination of resin (Unsaturated Polyester Resin) and single variable filler which is added at several addition levels. This viscosity experiment was designed and conducted to illustrate the differences in resin demand between standard fillers and biochar. The viscosity measurements collected during the study are shown below.

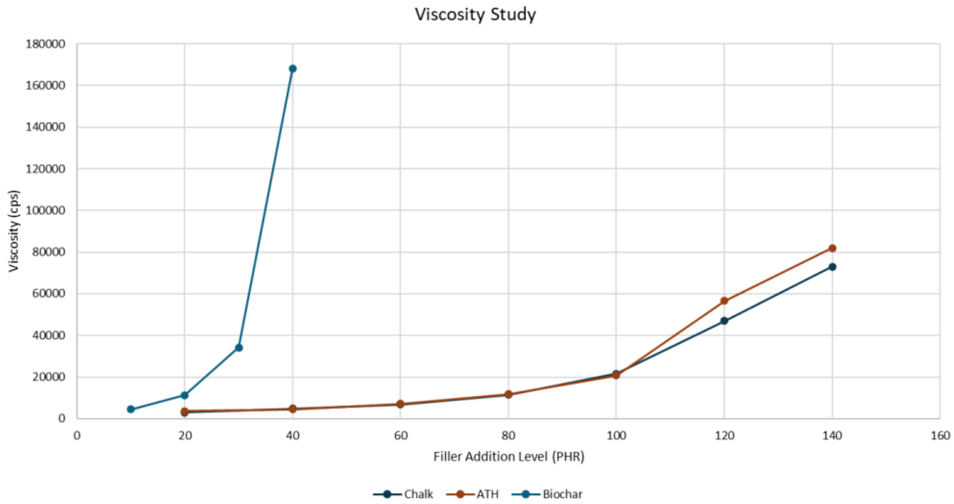

Figure 2. Viscosity measurements of URP mixed with fillers at different addition levels.

Examining the results of the study it is immediately evident that the biochar has a much higher resin demand than the chalk or ATH. This means that it will be challenging to add more than a small amount of the biochar to the formulation while still maintaining a low enough viscosity for fiber impregnation. The results show that the viscosity is able to remain relatively low for addition levels at 10, 20, and 30 PHR. However, the viscosity exponentially increases at 40 PHR. For chalk and ATH, it shows that the viscosity remains relatively low for addition levels up to 120 PHR which is 4x more than the biochar.

3.3.2.SMC Paste Formulations and Viscosity Experiment

Based on the initial results observed from the previous experiment some SMC paste formulations were developed for a basic low-shrink UPR formulation and a basic fire-retardant UPR formulation, as well as a variation of these formulations where a portion of the traditional filler is replaced with biochar. In this section viscosity measurements were collected on these prospective SMC paste formulations to ensure that the viscosity is maintained at acceptable levels for fiber impregnation during an SMC compounding run. Unlike the previous experiment these paste formulations will include multiple solid additives and closely mimic what was compounded.

Table 4. SMC Paste Formulations (in PHR) for the viscosity experiment.

Component	C-CA (PHR)	B-CA (PHR)	C-FR (PHR)	B-FR (PHR)
UP Resin	70	70	70	70
LPA	30	30	30	30
Calcium Carbonate	120	80	0	0
Thickener	2	2	2	2
Zinc Stearate	4	4	4	4
Biochar	0	20	0	20
ATH	0	0	120	80

The table above shows the formulations for the four SMC pastes mixed for the experiment. The C-CA and C-FR represent the control low-shrink and control fire retardant formulations respectively. The B-CA and B-FR represent the biochar filled low-shrink and fire-retardant formulations respectively. The table shows that the only difference in formulation between the control and biochar filled formulations is that the biochar filled formulations has replaced 40 PHR of the industrially standard filler (chalk for low-shrink and ATH for fire retardant) with 20 PHR of the biochar. Thus, the differences in the results observed between the control and biochar filled formulations show the impact of replacing 40 PHR of the industrially standard filler with 20 PHR of the biochar. The filler addition levels were selected to allow for a relatively significant amount of biochar to be added to the formulation while still maintaining low enough viscosity for processing.

The purpose of this viscosity experiment is to measure the expected viscosity of the SMC paste as the SMC is being compounded. The components of the pastes were mixed in the following order: Resin > LPA > Zinc Stearate > Chalk > Biochar > ATH > Thickener. Once mixed the viscosity of the samples were measured using a Brookfield DV2Plus Viscometer with t-bar spindles and helipath motor. The viscometer was set to collect measurements every 5 seconds over a period of 30 minutes. The results of these tests are displayed in the figure below.

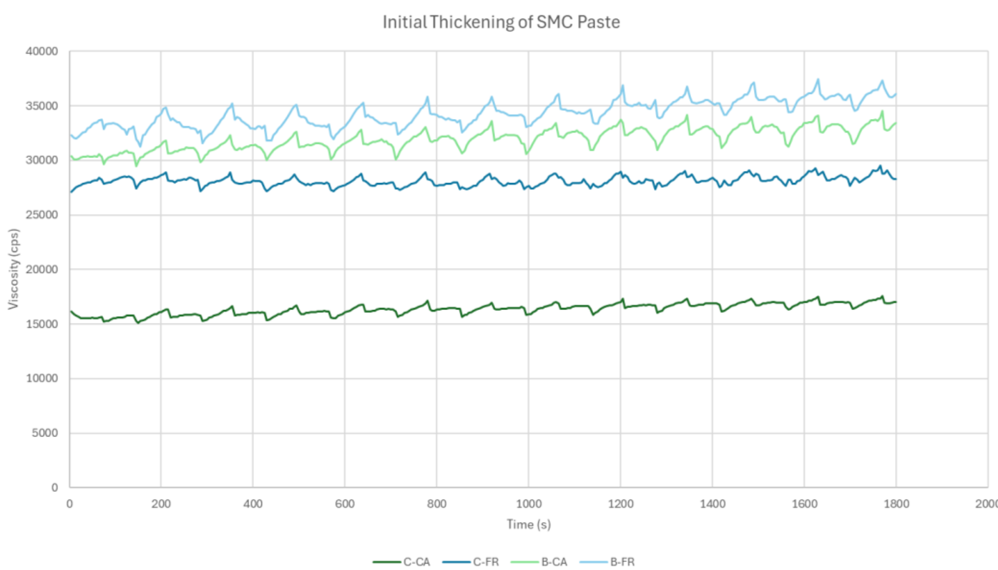

Figure 3. Viscosity measurements of SMC paste formulations for 30 minutes immediately after mixing.

The figure shows that the biochar filled SMC paste formulations have a higher viscosity than their control counter parts. This means that replacing 40 PHR of the industrial filler with 20 PHR of biochar will still result in a higher viscosity. The control low shrink formulation has the lowest initial viscosity out of all of the samples with the control fire retardant formulation having a higher viscosity (closer to that of the biochar filled formulations). All of the formulations have acceptable viscosity for processing.

3.4. SMC Compounding

With the confirmation that the viscosity of the SMC paste formulations would be acceptable for SMC compounding larger batches of the paste formulations were mixed (enough to make 12.5 kg of SMC). The SMC paste formulations vary only slightly from the samples made for the viscosity study, with added inhibitor and peroxide for curing. The paste formulation for the compounded SMC is shown in the table below.

Table 5. Paste Formulations (in PHR) for the compounded SMC.

Component	C-CA	B-CA	C-FR	B-FR
UP Resin	70	70	70	70
LPA	30	30	30	30
Calcium Carbonate	120	80	0	0
Thickener	2	2	2	2
Zinc Stearate	4	4	4	4
Biochar	0	20	0	20
ATH	0	0	120	80
pBQ (5%)	0.4	0.4	0.4	0.4
TBPB	1.5	1.5	1.5	1.5

Similar to the viscosity study samples the components were mixed in the following order: Resin > LPA > Peroxide > Inhibitor > Zinc Stearate > Chalk > Biochar > ATH > Thickener. All of the SMC batches were made with a target fiber content of 30% wt glass fiber. Once the SMC was compounded it was placed in a maturation chamber set at 33 ºC for 3 days and then kept at room temperature for 1 day prior to molding.

3.5. SMC Moudling

The SMC was compression molded in an 18-inch by 18-inch square plaque tool using a 2,500-tonne hydraulic compression molding press. The SMC was cut into charges roughly 38 cm by 38 cm. Two of these charges were required to mold a roughly 3 mm thick part. All of the parts were molded using the same press parameters. A table summarizing the press parameters is shown below.

Table 6. Summary of press molding parameters.

Parameter	Setting
Tool Surface Temperature (ºC)	145 ºC
Press Force (kN)	1,500 kN (72 bar)
Vacuum Time (s)	15 s
Cure Time (s)	120 s
Closing Speed	Ramp: 3 mm/s to 1mm/s over distance of 10 mm
Vacuum Time (s)	15 s

All of the material molded well (no obvious issues during molding). For each batch of material 6 replicate plaques were compression molded. A summary of the parts made for each batch of material is shown below. One important difference to note is the lower density of the biochar filled formulations in comparison to the control formulations.

Table 7. Summary of SMC plaques.

SMC Batch ID	Mass (g)	Thickness (mm)	Calculated Density (g/cm^3)
C - CA	1191 ± 11	3.12 ± 0.05	1.83 ± 0.03
B - CA	1117 ± 58	3.11 ± 0.15	1.72 ± 0.01
C - FR	1142 ± 26	3.07 ± 0.07	1.78 ± 0.01
B - FR	1107 ± 70	3.13 ± 0.21	1.69 ± 0.01

Figure 4. C-CA SMC part in mold.

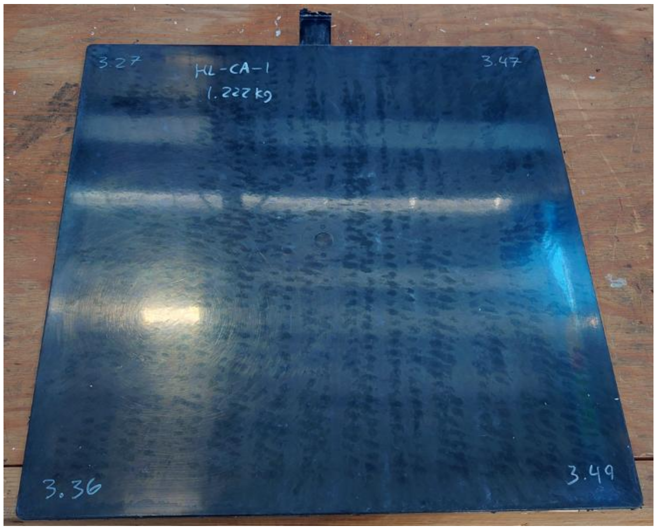

Figure 5. B-CA SMC part (previous naming convention HL-CA).

3.6. Sample Extraction

Three plaques from each batch were selected for sample extraction. The sample extraction follows the diagram shown below. Where the orange represents the tensile samples, the green represents the flexural samples, the purple represents the impact samples, and the blue represents the UL94 samples (for FR formulations only). All samples were extracted via waterjet cutting. The red dotted line shows where the plaque was cut prior to loading it into a waterjet cutter.

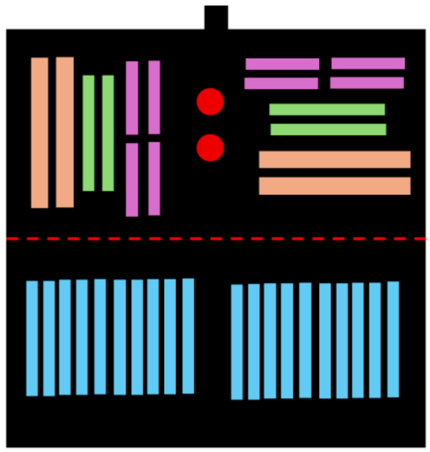

Figure 6. Plaque sample extraction plan.

4. Results and Discussion

This section details the tests which were conducted on the samples and their results as well as an analysis of the gathered data.

4.1. Tensile Testing

From each batch of SMC material three plaques were selected for sample extraction. From each plaque 4 tensile samples were extracted 2 were in the 0º orientation and 2 were in the 90º orientation to mitigate the influence of fiber alignment on the results. The tensile tests were conducted according to the standard ASTM D638. The results of the tensile tests are summarized in the figure below.

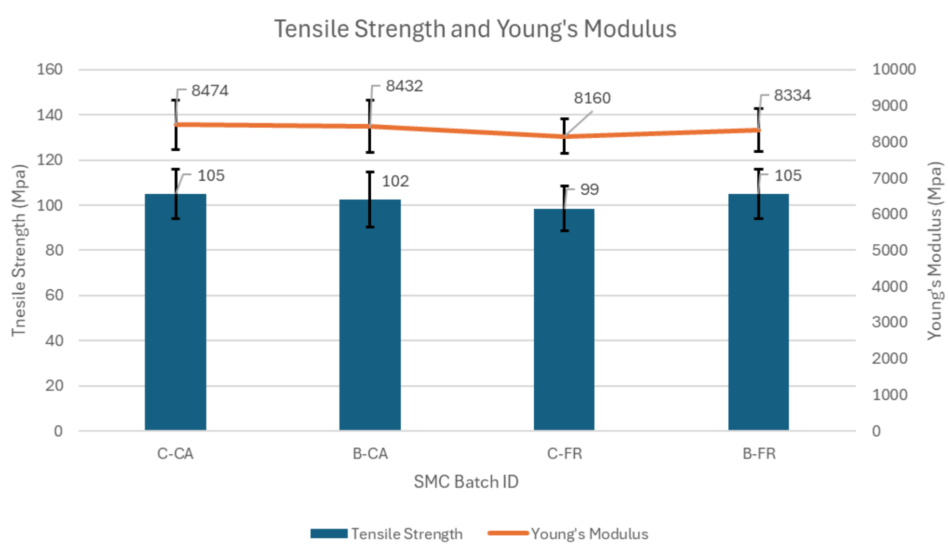

Figure 7. Summary of tensile test results.

The tensile test results show that there is very little difference between the control formulations and their biochar counterparts. This indicates that the addition of the biochar filler does provide any negative impact of mechanical properties when compared to the standard industrial fillers.

One interesting difference between the biochar and the traditional fillers is that the biochar has a significantly reduced density in comparison. The lower density of the biochar and the fact that the 40 PHR of industrial filler was swapped for 20 PHR biochar means that the biochar filled formulations have a lower density than the control formulations. Taking the difference in densities into account by examining the specific tensile strength and young's modulus can illustrate a potential advantage of the biochar SMC over the traditional fillers. The figure below shows the specific tensile strength and young's modulus of each material formulation.

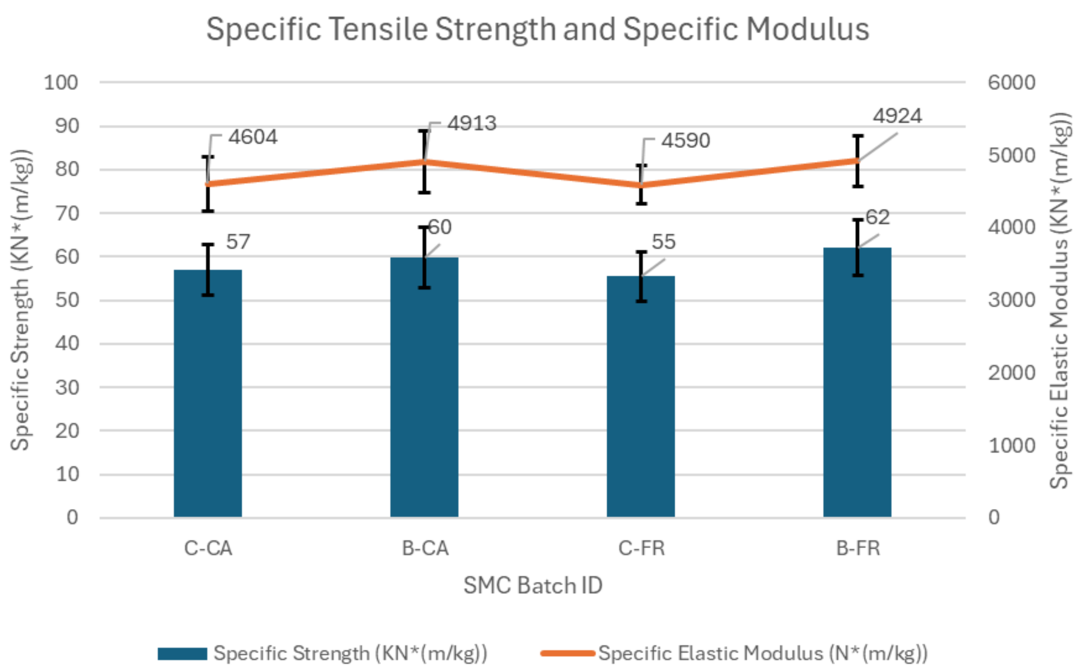

Figure 8. Specific tensile strength and young's modulus.

Looking at the specific tensile strength and young's modulus showcases the benefits of the lower density of the biochar filled material. The differences between the control and biochar filled material are only slight, but both biochar filled formulations perform better in terms of specific strength/modulus compared to the control formulations.

4.2. Flexural Testing

From each batch of SMC material three plaques were selected for sample extraction. From each plaque 4 flexural samples were extracted 2 were in the 0º orientation and 2 were in the 90º orientation to mitigate the influence of fiber alignment on the results. The flexural tests were conducted according to the standard ASTM D790. The results of the flexural tests are summarized in the figure below.

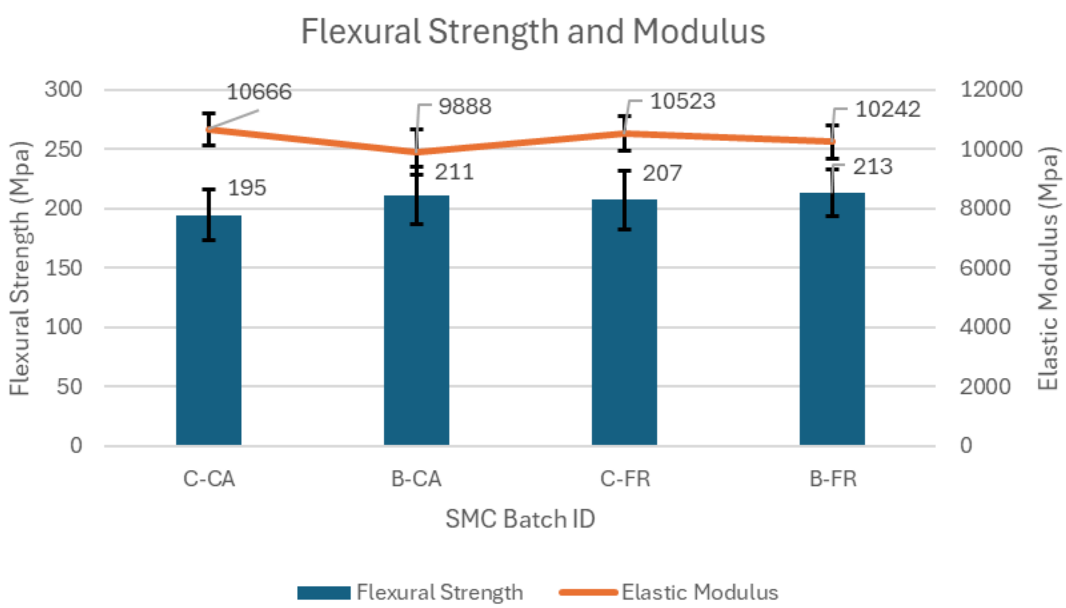

Figure 9. Summary of flexural test results.

The flexural test results follow a similar trend where the control and biochar filled formulations test similarly. The biochar formulations generally appear to have a slightly reduced modulus compared to the control formulations. However, the biochar formulations have a slightly increased tensile strength compared to the control formulations.

Once again, the specific strength and modulus should be examined to see how the materials perform relative to their densities. The figure below shows the specific flexural strength and young's modulus of each material formulation.

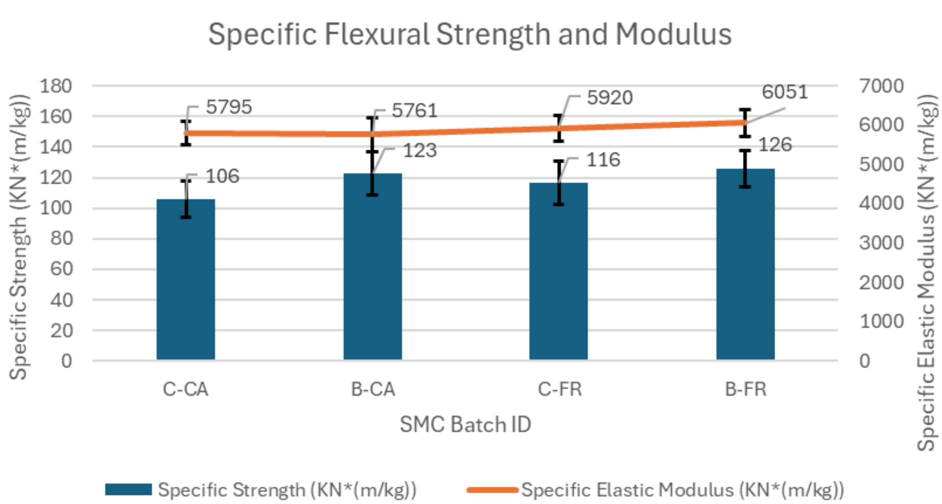

Figure 10. Specific flexural strength and modulus.

The results follow a similar trend to the tensile test results. The specific flexural strength of the biochar filled formulations is more significantly higher than what was observed for the tensile strength. However, the flexural modulus of the biochar materials are more similar to the control formulations. Overall, the specific flexural strength/modulus of the biochar filled materials in an improvement over the control formulations.

4.3. Impact Testing

From each batch of SMC material three plaques were selected for sample extraction. From each plaque 8 flexural samples were extracted 4 were in the 0º orientation and 4 were in the 90º orientation to mitigate the influence of fiber alignment on the results. The impact tests were conducted according to the standard ASTM D256 (notched Izod). The impact strength of each batch of SMC is shown in the figure below.

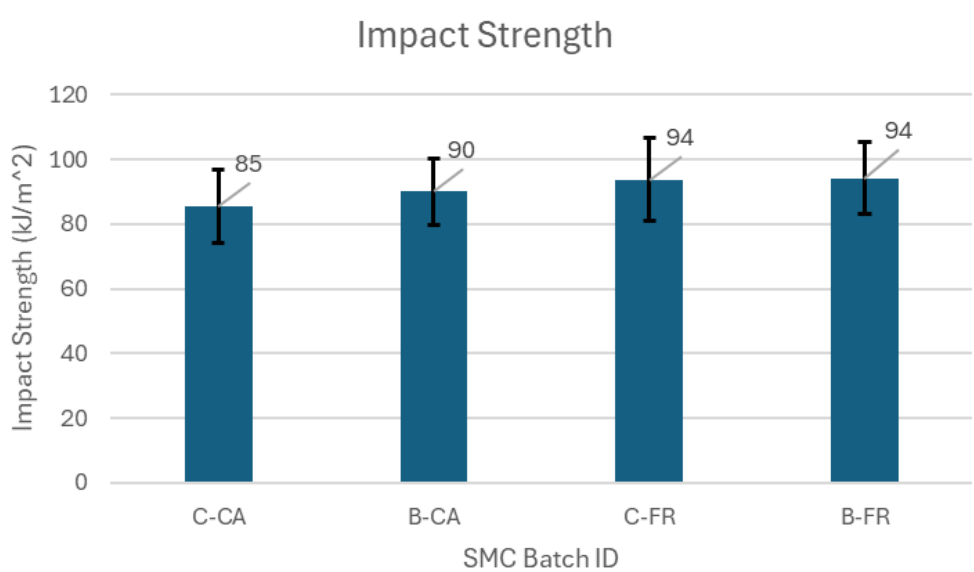

Figure 11. Impact strength of each SMC batch.

There's a slight increase in impact strength of the biochar filled low-shrink SMC compared to the control low-shrink formulation. The results for the control FR and biochar FR formulations match very closely. The testing results suggest that biochar is not having a significant effect on the impact strength of the material and could potentially provide a slight benefit.

4.4. Flammability Testing

This section reviews TGA tests conducted on the fillers used in the FR formulations (ATH and biochar) and UL94 tests conducted on the control and biochar filled SMC.

4.4.1. Thermogravimetric Analysis (TGA)

TGA tests showcase a materials thermal stability by measuring the mass of the material prior to heating and then periodically measuring the mass as the material goes through a heating program.

During the test the material's degradation temperature can be observed and the amount of the material remaining after thermal degradation.

In this experiment the fillers used in the fire-retardant formulations (ATH and biochar) are tested using TGA to understand their thermal stability. The parameters of the TGA test are shown in the table below. The results of the test are shown in the figure below.

Table 8. Summary of TGA experiment parameters.

Parameter	Setting
Moisture Removal	25 ºC to 107 ºC (ramp 17 mins, hold 15 mins)
Burn Off	107 ºC to 565 ºC (ramp 22 mins, hold 2 hours)

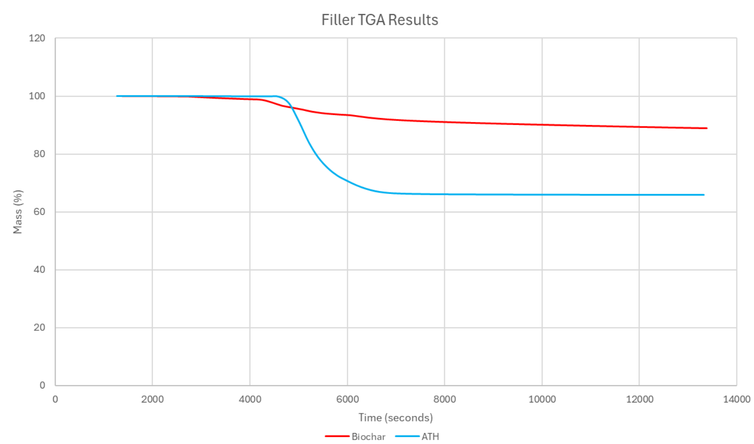

Figure 12. TGA results for fillers used in FR formulations.

The results from the graph show that the biochar is quite thermally stable even at the elevated temperature of 550 ºC it only degrades slightly losing about 10% of it's total mass. Polymers are known to thermally degrade much more than what is seen with biochar, thus the relatively inert biochar can provoide some FR benefit over the polymer. The ATH degrades a significant amount which is to be expected and is the source of it's FR properties. It is well established that ATH decomposes endothermically and releases water during this process. The combustion reation of ATH is shown below [6].

$$2Al(OH)_2 \rightarrow Al_2O_3 + 3H_2O\ (1050\frac{kJ}{kg})$$

The endothermic decomposition absorbs energy, the water produced is non-flammable and can help extinguish the flame, and finally the Al_2O_3 forms a thermally insulating coating [6]. Although the biochar is relatively inert it does not provide the same FR benefits as the ATH.

4.4.2. UL 94 Testing

UL 94 testing helps categories a plastic or composites: flammability, likelihood to spread fire, and ability to self extinguish. The testing setup is relatively simple a sample is placed above a flame for a period of time and removed from the flame the duration of the burning of the sample is recorded (T1) if the sample stops burning it is placed in the flame again and removed once more and again the duration of the burning is recorded (T2). If the flame is extinguished again then the afterglow time of the sample is recorded (T3). When the sample is removed from the flame it is placed above a cotton pad and if the sample drips while burning it is recorded, and if the dripping sample ignites the cotton pad it is recorded. Depending on how the sample performs during the test it is placed under a certain classification. The classifications provide a simple method of quickly understanding how the sample performs during the test. The classifications are as named the following in order of least fire retardant to most fire retardant: V2, V1, and V0. If a sample demonstrates no ability to resist burning it is not classified by the test and does not meet any classification.

To conduct the test two sets of samples are required for each material being tested. One set of samples undergoes conditioning of 23 ºC and 50% humidity for 48 hours, and the other 5 were conditioned at 70 ºC for 7 days. In this experiment the control fire retardant formulation and the biochar filled fire retardant formulation are evaluated. The table below shows the UL94 test results for samples conditioned at 23 ºC.

Table 9. UL94 test results for samples conditioned at 70 ºC for 7 days.

Sample #	Thickness (mm)	T1 (s)	T2 (s)	T3 (s)	Dripped	Ignited Cotton	Burned to Clamp	UL94 Rating	Sample #
C-FR-6	3.04	0	0	0	N	N	N	V0	C-FR-6
C-FR-7	3.06	0	1	0	N	N	N	V0	C-FR-7
C-FR-8	3.05	0	1	0	N	N	N	V0	C-FR-8
C-FR-9	3.04	0	1	0	N	N	N	V0	C-FR-9
C-FR-10	3.04	0	1	0	N	N	N	V0	C-FR-10
B-FR-6	3.02	0	2	0	N	N	N	V0	B-FR-6
B-FR-7	3.02	0	1	0	N	N	N	V0	B-FR-7
B-FR-8	3.02	0	2	0	N	N	N	V0	B-FR-8
B-FR-9	2.99	0	7	0	N	N	N	V0	B-FR-9
B-FR-10	3.00	0	2	0	N	N	N	V0	B-FR-10

From the UL94 testing it can be shown that the biochar filled samples did not have the same level of fire resistance compared to the control samples. The fire-retardant mechanism of ATH and the TGA test results for the biochar align with this finding. Replacing 40 PHR of ATH with 20 PHR of biochar will have a negative impact on the material's fire resistance.

5. Conclusion

This paper describes the work conducted to create two biochar filled SMC formulations which are filled with a combination of biochar and industrially standard SMC fillers and two control formulations which use only industrially standard fillers. Biochar filler presented two primary processing challenges: high resin demand and high moisture content. Viscosity studies were conducted to narrow down appropriate addition levels for biochar to ensure good the SMC paste was processable. Based on the results of the viscosity study it was decided that 40 PHR of the industrially standard filler can be replaced with 20 PHR biochar while maintaining a low viscosity for good processability. Two basic SMC formulations were examined for the test: low-shrink and fire-retardant formulations. A drying procedure was developed for the biochar filler to ensure the moisture content was low for processing (mitigate impact on viscosity build and blisters during molding).

The preparations made to compound biochar filled SMC were successful and allowed for the compounding of two varieties of biochar filled SMC which matured and molded very similarly to their control formulation counterparts. The molded SMC plaques were cut into mechanical test specimen and the properties of the biochar filled SMC formulations were compared to their control counterparts. The mechanical testing results showed that the biochar filled composites had a slightly better specific tensile/flexural properties. Essentially the biochar formulations were found to maintain similar mechanical properties while having a lower density than the control formulations. The differences were relatively minor; however, the results indicate that the addition of biochar can provide benefits for SMC formulations. The flammability of the materials were evaluated using UL94 testing. The UL94 testing indicated that the biochar filled samples were more flammable than the control formulations. There appear to be methods by which biochar filler can provide some FR benefit, however, based on the results of this experiment, any FR benefits provided by the biochar were outweighed by the loss of the 40 PHR ATH required to incorporate the biochar into the formulation.

6. Future Work

This paper outlines the experiments conducted on the very first biochar filled SMC to the authors knowledge. There are several ways that these initial experiments can be expanded on in subsequent experiments. Biochar is known to be a “tunable” material, meaning different morphologies of biochar can be achieved by using different bio-mass feedstocks and different pyrolysis parameters. These different morphologies can be more effective for specific applications. Sourcing biochar from different suppliers who use different feedstocks and different pyrolysis parameters and evaluating the resulting SMC in the same methods can help identify the impact of the different morphologies of biochar on SMC properties. In this experiment biochar was only added at 1 addition level, different addition levels of biochar should be explored to more clearly understand the relationship between the filler and SMC properties.

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