

BIODEGRADABLE COMPOSITE HYDROMULCHES FOR SUSTAINABLE
ORGANIC HORTICULTURE

by

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of

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NOMENCLATURE

LDPE = low density polyethylene

HM = hydromulch

C:N = carbon to nitrogen ratio

MPa = megapascals

N = newton

USDA = United States Department of Agriculture

mm = millimeters

min = minutes

cm = centimeters

Kg/ha = kilograms per hectare

lb./ac = pounds per acre

AURI = Agricultural Utilization Research Institution

GG = guar gum

PH = psyllium husk

CM = camelina meal

P%GG = paper and guar gum where % represents % of guar gum by weight (2,4 or 6)

P%PH = paper and psyllium husk where % represents % of psyllium husk by weight (2,4 or 6)

P%CM = paper and camelina meal where % represents % of camelina meal by weight (2,4 or 6)

W%GG = wood and guar gum where % represents % of guar gum by weight (2,4 or 6)

W%PH = wood and psyllium husk where % represents % of psyllium husk by weight (2,4 or 6)

W%CM = wood and camelina meal where % represents % of camelina meal by weight (2,4 or 6)

NOMENCLATURE CONTINUED

W%CM = wood and camelina meal where % represents % of camelina meal by weight (2,4 or 6)

#PH%GG = paper and hemp blend where # represents the amount of paper by weight (75 or 50) and guar gum where % represents % of guar gum by weight (2,4 or 6)

#PH%PH = paper and hemp blend where # represents the amount of paper by weight (75 or 50) and psyllium husk where % represents % of psyllium husk by weight (2,4 or 6)

#PH%CM = paper and hemp blend where # represents the amount of paper by weight (75 or 50) and camelina meal where % represents % of camelina meal by weight (2,4 or 6)

#PW%GG = paper and wood blend where # represents the amount of paper by weight (75 or 50) and guar gum where % represents % of guar gum by weight (2,4 or 6)

#PW%PH = paper and wood blend where # represents the amount of paper by weight (75 or 50) and psyllium husk where % represents % of psyllium husk by weight (2,4 or 6)

#PW%CM = paper and wood blend where # represents the amount of paper by weight (75 or 50) and camelina meal where % represents % of camelina meal by weight (2,4 or 6)

RFI = rain fastness index

gsm = grams per square meter

CV = coefficient of variation

LSD = least significant difference

ABSTRACT

In agriculture, mulch helps retain soil moisture and temperature while preventing weed growth. The most common material used for commercial mulching is low-density polyethylene (LDPE). At the end of the growing season, this plastic is typically buried or burned, causing a negative impact on the environment. This project aims to develop an alternative to LDPE mulch that is acceptable for organic farming and biodegradable. The tested hydromulch (HM) treatments contain a mixture of paper pulp, wood fiber, or hemp hurds combined with a tackifier and water. The tackifiers evaluated were guar gum, psyllium husk, and camelina meal, at various concentrations. These treatments were tested for tensile strength, puncture resistance, rain fastness, density, soil adhesion, porosity, and C:N ratio. The results have shown that samples containing tackifiers outperformed the control that contained no tackifier in the strength tests but not in the rain fastness or soil adhesion tests. Paper was the best fibrous material and guar gum was the top performing tackifier. When tackifier blends were considered, an interaction between two tackifiers occurred resulting in a decrease in strength. Blends containing wood fiber and hemp hurds did not show promising results. The puncture resistance of all mulches significantly decreased at 50% moisture level regardless of tackifier type. Some formulations performed well and could be promising in future field trials. The next step will be to examine these formulations outdoors in large-scale field studies.

INTRODUCTION

1.1 Motivation

The use of mulch in horticulture production is essential. Mulch helps suppress weeds, enhance soil nutrient levels, reduce water evaporation rate (Kader et al., 2017), and control soil temperature, (Lopez-martin et al., 2021). The most widely used mulch material is low density polyethylene (LDPE) sheet mulch (Mas et al., 2021). Mulch can increase yields by up to 100-200% by regulating soil temperature and suppressing the growth of weeds (Salakangas et al., 1973). The problem with LDPE is that it is not commonly recycled at the end of the grow season (Moore and Wszelaki, 2016) because it is too dirty (Chen et al., 2018). Therefore, LDPE is more commonly burned or buried, both harmful to the surrounding ecosystem. Studies into this matter have found, even after removal, traces of the LDPE sheets in soil, in nearby water sources, and even in humans (Lopez-Marin et al., 2021). To overcome this issue many studies have been done to create a cellulose-based product that is biodegradable but still performs similarly to LDPE. One possible solution are biodegradable cellulosic-based liquid applied mulches known as hydromulches (HM). Organic, biodegradable HMs are typically composed of fibrous cellulosic materials such as paper pulp, cotton, or straw, mixed with a small amount of tackifier such as guar gum to help the fibers adhere to one another as well as the soil. These dry ingredients are then mixed with water, allowing the HM to be sprayed onto the soil and once dry, leaving a hardened mulch layer. Ideally the HM will break down and decompose into the soil at the end of the growing season. Unlike LDPE, this product does not need to be removed from the soil and disposed of, eliminating additional labor (Salakangas et al., 1973).

Previous work has looked at fibrous materials such as recycled paper, cardboard, thermally treated fiber, wheat straw, used mushroom substrate, rice husk, rice bran (Claramunt et al., 2019), and cotton (Holt et al., 2005). These materials compromise the bulk of the HM formulation at around 96% of the overall dry weight (Vaughn et al., 2012). These materials are sometimes used alone with a tackifier, and sometimes a combination of multiple is used. Tackifiers used include xanthan, high amylose NAPAC, psyllium, lignin, camelina seed meal, lesquerella seed meal, guar, pectin, methyl cellulose, low amylose NAPAC, and waxy starch (Vaughn et al., 2012). Currently the most commonly used is guar gum. This has motivated work into alternatives as guar gum has seen price increases as high as 800% due to increased use in the oil industry and limited global production (Vaughn et al., 2012).

Strength is a critical property in the performance of a HM. More specifically, puncture resistance allows for inferences to be made about HM's ability to suppress weed growth. One study done by Verdú et al. found a range of puncture resistances from different formulations with most ranging from 0.4 to 1.4 MPa. The best performing HM contained paper, wheat straw, gypsum powder, and kraft paper pulp with a puncture resistance of 1.47 MPa. (Verdú et al., 2020).

Another important property that is often tested is rain fastness. That is, the amount of material washed off or lost during a rain event. A study was conducted by Vaughn et al. to compare different tackifier materials. The results showed that the HM formulations maintained 75.2% (waxy starch) and 91.7% (xanthan) of their original weight. Guar gum was somewhere in between at 0.880 (Vaughn et al., 2012). Another study done by Holt et al., tested mulch materials including cotton byproducts (COBY), rice hulls, cottonseed hulls, paper, and thermally treated

wood fiber. It was found that the best performing material was rice hulls with material loss of 1.5% original weight.

Few effective herbicides are available for organic horticulture. Additionally, herbicides can be difficult at the beginning of a crop's life cycle making mulch very important to suppress the emergence and growth of weeds that can take away water and nutrients from the crop (Mas et al., 2021). In one study, wheat straw, rice husk, and used-mushroom substrate were used as HM and applied to soil containing weed rhizomes. Results showed a significant difference in weed emergence rates of rhizomes that had a HM treatment (around 20%) versus rhizomes that did not have HM (around 85%) meaning the HM successfully suppressed many of the rhizomes (Mas et al., 2021).

A study was done by O'Brien et al. to see how adding HM at different application rates affects soil temperature and evaporation. It was found that the 1x rate reduced evaporation by 16% and 3x reduced evaporation by 32% compared to 0x (O'Brien et al., 2018).

1.2 Objectives

Work done at Montana State University (MSU) focused on testing the properties of different organic, biodegradable hydromulch formulations. The goal was to compare different organic HM formulations and determine the top performers through mechanical properties tests. These tests included tensile strength, puncture strength, rain fastness, soil adhesion, density, porosity, and C:N ratio. In addition, puncture resistance was tested at various moisture levels, and puncture resistance was done before and after the rain fastness test. The organic materials used for these formulations included recycled paper, thermally treated wood fiber, and hemp hurds in combination with guar gum, psyllium husk or camelina meal as tackifier. Both fiber

types and tackifier types were also combined. Based on previous work, it was hypothesized that the paper formulations would perform the best. Ultimately, the top performers will continue to be tested in field trials.

LITERATURE REVIEW

2.1 Background

The benefits of mulching have been known since the early 1920's. Mulches are materials placed on the soil surface to form a physical barrier that limits evaporation, controls weeds, maintains soil structure, and protects crops from contamination (Kasirajan et al., 2012). No perfect mulch material exists. Crop type, climate, and many other factors can all play a large role in determining the perfect mulch (Kader et al., 2017). Currently, low-density polyethylene (LDPE) mulches are less expensive and when compared to organic mulches. In contrast, organic HM's reduce labor needing for weeding, and provide nutrients to the soil after decomposing for future crops (Kader et al., 2017). This review will discuss LDPE, biodegradable mulch films, and hydromulch in relation to research results associated with each mulch type.

2.1.1 Polyethylene Mulch

LDPE mulch has been used in the agricultural industry for vegetable and fruit production for around 70 years. Polyethylene as a plastic film was developed in 1938. Shortly after that, in the early 1950's, the agricultural industry adopted the material as farmers quickly discovered how much they could improve their yields (Lamont et al., 2017). Today, LDPE remains the most commonly used mulch material for large scale fruit and vegetable cultivation. In studies dating back to the 1950's when LDPE mulch use began, farmers tested other alternatives, but none could compete with the cost and performance of LDPE. However, due to recent global sustainability efforts, LDPE is now more than ever under scrutiny for its environmental impacts when used in large quantities as a mulch material.

One of the most important characteristics of mulch other than weed suppression and evaporation control is its ability to buffer soil temperatures. In hotter climates the mulch helps reduce the soil temperature and in colder climates, it helps to maintain warmer soil temperatures allowing crops to be grown in less-than-ideal climates. An example of this is the growth of pickling cucumbers in Finland. Pickling cucumbers require 10-12 degrees C to grow. When the seeds are planted at the beginning of the growing season, the temperatures are often lower than this which would typically result in a drastic drop in yields and even crop loss, but due to the use of LDPE mulch, the yields are increased by up to 200% (Salakangas et al., 1973).

To apply LDPE mulch, the farmer must first invest in specialized equipment. This equipment includes a bed press, mulch layer, mulch transplanter, and mulch itself (Lamont, 2017). These costs significantly increase the cost of production for a specific crop. However, they are worthwhile and quickly paid off given the increased yield and reduction of herbicides and water needed. In conclusion, LDPE remains the most used mulch material, despite the negative impacts it has on the environment.

2.1.2 Biodegradable Films

An alternative to LDPE is biodegradable plastic films. Biodegradable films are prepared as sheets and laid out onto the soil, similarly to LDPE application. Some materials that are used for this application include Mater-Bi© and Biofilm© as well as poly (butylene adipate-co-terephthalate) (PBAT), under commercial name Ecoflex© 7011 along with pigment Sicopal Black© K0095 (Bilck et al., 2010). One study looked at this biodegradable PBAT film and compared it to LDPE in multiple different tests. These tests included max tensile strength, elongation at break, and grammage.

After five weeks, Bilck et al. saw the PBAT films began showing cracks with some as early as two weeks while the LDPE showed no change after eight weeks. These cracks allowed for weeds to easily emerge. Before application, tensile strengths and elongation at break were not significantly different between the biodegradable PBAT film and the LDPE. However, as one would expect, after the biodegradable film was exposed to the elements for eight weeks, they lost around 50% of tensile strength while the LDPE lost 20%. No significant difference was seen in elongation at break.

Finally, the grammage was tested. This test measures the amount of material per square meter and compares the initial value to the value after eight weeks showing the rate at which the material biodegrades. As expected, there was little to no loss in grammage for the LDPE; however, for the PBAT material, the grammage decreased by 36%. In conclusion, the experiment found that the PBAT films performed similarly to the LDPE. Although the strength and grammage decreased over the eight weeks, the properties did not deteriorate enough to affect the crop yield.

Another study was done by Cirujeda et al. comparing oxo-degradable plastics commercially available as Mater-Bi®, Biofilm®, Enviroplast®, MimGreen®, and Saikraft®. Biofilm® was unsuccessful in suppressing weeds due to weeds puncturing through the plastic whereas other plastics did not have this problem. The material was prone to tearing if applied in the wind. All other materials including LDPE suppressed 87-100% of weeds. All treatments yielded significantly higher yields than the control (untreated bare soil). The increase in yield was as high as 78% with the addition of plastic mulch. However, the section of biodegradable plastic buried under the soil did not biodegrade since it was not exposed to oxygen. This study

concluded that although more expensive, many biodegradable alternatives are sufficient for suppressing weeds and increasing yields. For many farmers, the cost is the most crucial factor. Since biodegradable plastics do not increase yields compared to LDPE, they cannot afford to switch to a more expensive material. Additionally, no available biodegradable plastics are approved for organic horticulture production at this time. Additionally, there have been negative effects associated with the polymer components of biodegradable films on plant productivity and soil microorganisms (Serrano-Ruiz et al., 2021).

2.1.3 Hydromulch

The previously mentioned materials may work and are biodegradable but are not approved for organic farming. Cellulose-based hydromulch consists of organic materials that will not only biodegrade but also allow for USDA organic approval. A hydromulch is a slurry consisting of a cellulosic fibrous material such as paper pulp, cardboard, thermally treated wood fiber, wheat straw, used mushroom substrate, rice husk, rice bran (Claramunt et al., 2019), and cotton (Holt et al., 2005) as well as a tackifier. Tackifier options include guar gum, plantago or psyllium husk, xanthan gum, seed meals such as camelina meal, or other starches like corn starch (O'Brien et al. 2018). Water is added to the mixture to create a slurry that can be sprayed over bare soil creating a biodegradable layer of natural material once dry. HM is commonly used for erosion control and restoration (Faucette et al., 2006) but is much less common in horticulture production.

2.1.3.1 Hydromulch Testing Methods One study examined different HM formulations' puncture strengths using a universal testing apparatus to lower a 7.86 mm diameter probe at a

speed of 4 mm/min down onto a sample of HM and compare the peak values of different HM formulations and LDPE (Verdú et al., 2020). All formulations that were tested were paper-based with the addition of some other natural ingredients. The average puncture strength value from the HM formulations was 1.47 MPa. This value is much lower than that of LDPE which was found to be 3.87 MPa. However, it has been found that most weeds will not penetrate when the HM puncture resistance exceeds 0.5 MPa (Verdú et al., 2020). The best formulation was found to be paper with wheat straw and white gypsum. However, these were conducted in a lab setting and the samples were dry. Because the HM will be exposed to various weather conditions it is important to know if moisture will affect the puncture resistance enough to allow for penetration.

Using herbicides can be very difficult at the beginning of a crop's life cycle making mulch very important to suppress the emergence and growth of weeds that can take away water and nutrients from the crop (Mas et al., 2021). Therefore, a study examined the weed emersion of crops treated with paper HM. Claramunt et al. (2020) found that paper HM could prevent 95.9% of weeds from emerging. A different study was done by Mas et al. (2021) that looked at the effectiveness in suppressing the emergence and continued growth of *Paspalum dilatatum*, *Cynodon dactylon*, and *Sorghum halepense* weed varieties with an HM. Wheat straw, rice husk, and used mushroom substrate were used as HM base materials and HMs were applied to soil containing weed rhizomes. Results showed that 20% of rhizomes emerged that had an HM treatment while 85% of rhizomes emerged from the bare soil control (Mas et al., 2021).

Because most HMs are made up of organic based, biodegradable material, they would be expected to break down quickly. Many different studies have shown that by the end of the growing season, little HM material remains. One study found that the mulch lost 300% of its

initial strength in ten days (Scarascia-Mugnozza et al., 2018). One might think this is a big problem but, the same study found that the degrading material continued to suppress weeds and crop yield was not affected.

Another important property that is often tested is rain fastness, which is the amount of material washed off or lost during a rain event. A study done by Vaughn et al. (2012) simulated a rain event of 1.6 cm rainfall over 30 min to compare different tackifier materials. This study used commercially available Hydrostraw® that contained no tackifier. Tackifiers were then added at a rate of 4% by weight. The results showed that the HM formulations maintained 75.2% (waxy starch) and 91.7% (xanthan) of their original weight. Guar gum was somewhere in between at 0.880 (Vaughn et al., 2012). Another study done by Holt et al. (2005), used a similar procedure but tested mulch materials including cotton byproduct (COBY) (Holt et al., 2003), rice hulls, cottonseed hulls, paper, and thermally treated wood fiber. The best performing material was rice hulls, with a material loss of 1.5% original weight. The worst material was thermally treated wood fiber, with a loss of 8%, with other materials in between at around 5% loss (Holt et al., 2005).

A study was done by O'Brien et al. to see how adding HM at different application rates affects soil temperature and evaporation. Commercially available wood fiber-based HM was applied at 0x, 1x and 3x manufacturer recommended application rate. Over 39 days, temperature fluctuations were smaller with the 3x rate than the 1x, and the 1x fluctuations were smaller than the 0x rate. Additionally, it was found that the 1x rate reduced evaporation by 16% and 3x reduced evaporation by 32% compared to 0x (O'Brien et al., 2018).

2.2 Discussion

Some form of mulch is necessary for many agriculture types to maximize yield and profits. The most used and most available LDPE is not sustainable and does not align with the goals of organic farming bringing forth the need for a sustainable alternative. Biodegradable films may solve disposal issues associated with LDPE but are still not approved for organic farming, making HM an obvious potential solution. At least when dry, most HM formulations can be more than strong enough to suppress weeds which was confirmed by a study finding the addition of HM reduced the number of emerging weeds by 65%. Degradation is a major concern considering HM will begin biodegrading immediately after application. Just 30 min of rain can cause up to 8% of material loss. However, with suitable materials this value can be as low as 1.5% and it may be possible to reduce losses even further. Finally, the associated cost is one of the most significant issues with HM. HM is more expensive than LDPE. However, another consideration for HM is the reduced labor required. This is important because it is estimated LDPE removal costs ~\$250 per hectare (Warnick et al., 2006). Since it is sprayed onto the soil, a hydroseeding tractor is all that is needed. In addition to the reduced labor needed for the application, like the other biodegradable materials, nothing needs to be done at the end of the growing season to remove and dispose of the mulch. Biodegradable HM may be an alternative to LDPE able to be used by organic farmers. Further testing into various materials may help reduce costs and improve mechanical properties.

MATERIALS AND METHODOLOGY

3.1 Materials

The HM samples used in this study were prepared to achieve an application rate of 4483 Kg/ha (4000 lb./ac), a common rate used for HM application (Vaughn et al., 2012). The original formulations tested consisted of a cellulosic fibrous mulch base material and combined with plant-based tackifiers at varying concentrations (Table 1). Eventually, the mulch materials and tackifiers were blended to maximize mechanical properties and reduce overall cost of the HM.

U-Haul packing paper was chosen for this project because it was easily attainable in large quantities and made entirely of cellulosic material. At the end of the project, paper from an alternative source was tested for tensile strength and puncture resistance and compared to the values collected with the U-Haul paper. The thermally treated wood fiber was donated to Montana State University (Profile, Buffalo Grove, IL). In addition to the wood fibers, the product also contained a green dye to help with the application process. This dye was not organic but because this was the only thermally treated wood fiber available, it was assumed this dye would not impact the mechanical properties of the HM. A Montana based company Ind Hemp supplied the hemp. Anthony's organic guar gum and psyllium husk were both purchased in very fine powder from an online retailer (Amazon, Seattle, WA). Camelina meal was provided by the Agricultural Utilization Research Institute (AURI) located in Crookston, MN. The materials used to compose the different HM formulations are shown in Figure 1.

Guar gum prices have increased recently by as much as 800% (Vaughn et al., 2012). Because of this, guar gum was blended with a cheaper alternative, camelina meal, in hopes of maintaining mechanical properties while decreasing the overall cost of the formulation. Another

expensive product, psyllium husk, was also blended with camelina meal. The tackifiers were blended in HM at rates of 50/50 and 75/25 guar/camelina and psyllium/camelina by weight.

To simplify discussing the formulations used they were assigned abbreviations. The first characters denote the fibrous material used: P for paper, W for wood, and H for hemp hurds. In the cases where fibers were blended the abbreviation begins with a number such as 75 or 50 followed by the fibrous materials that were blended. The 75 or 50 represents the percentage of paper. The final part of the abbreviation represents the concentration of tackifier (2%, 4% or 6%) followed by the type of tackifier. GG is for guar gum, PH for psyllium husk, and CM for camelina meal. For example, P2GG represents paper + 2% guar gum. 75PW4CM represents 75% paper/ 25% wood fiber + 4% camelina meal.



Figure 1 – Materials used as tackifiers in the different HM formulations (top) and fibrous materials used (bottom)

Table 1 – Fibrous materials, tackifiers, and tackifier concentrations used for HM formulations

Fibrous Material	Tackifiers	Tackifier Concentrations
Paper Pulp	Guar Gum	2%
Thermally Treated Wood Fiber	Psyllium Husk	4%
Hemp Hurds	Camelina Meal	6%

3.1.1 Chemical Composition of HM Materials

To fully understand mechanical and physical behavior of the different HM formulations tested it is important to understand the chemical composition of the different fibrous materials used. The first material considered is paper pulp. Paper pulp is made by mechanically and chemically treating fibers to separate the cellulose (Davidsdottir, 2013) which are then used to make paper. Paper contains 90-99% cellulosic fibers which make up a network of self-bonding cellulose fibers that give paper its chemical and physical properties. The chemical structure of cellulose is β -anhydroglucose (Sahin, 2008). Wood fibers as well as hemp hurds also contain cellulosic fibers, but they are entrapped by woody outer layer called lignin. Lignin prevents the network of self-bonding cellulose fibers that occurs with paper making it more difficult to get the fibers to adhere (Shao et al., 2006). Additionally, when thermally treating wood fiber, the fibers lose strength and become brittle.

Tackifiers play a big role in HM as they act as the ‘glue’ that holds the fibrous materials together as well as to the soil. Guar gum molecules contain hydroxyl groups resulting in hydrogen bonding with cellulosic material (Mudgil, 2011). Psyllium husk has gel forming abilities when mixed with water due to mucilage found in the cells of the outer layer of the plantago seed. This gel mixes with the fibers in HM and acts as a tackifier once dry (Cui et al.,

2011). Camelina meal consists of 13% residual oil making it a promising tackifier for repelling water. Camelina meal also contains 30% crude protein (Günç et al., 2020). These proteins are like those found in canola meal which has shown potential as an alternative to petroleum-based adhesives. These proteins include albumin, globulin, and glutelin which shows the best adhesion properties (Berti et al., 2016).

3.2 Methodology

3.2.1 Preparation of Samples

The first part of the project was developing a method to create HM samples that could be repeated easily and consistently throughout the duration of the project. The chosen method was to mix the dry ingredients with water using an immersion blender and then pour the slurry over a very fine, nylon mesh screen like that used for making paper. The immersion blender, shown in Figure 2a, was used because it allows the operator to chop up the ingredients and mix them simultaneously in a couple of minutes. Nylon mesh screen (Figure 2b) was used as it allows the water to slowly drain from the slurry, similar to how the soil would ‘wick’ moisture from the mulch. The mesh is fine enough to prevent excess loss of mulch material. This is representative of what is observed when the HM slurry is applied to soil. The amount of water added was determined by adding dry ingredients to a bucket and adding water until the optimum amount was found so that the immersion blender could easily combine the ingredients and the slurry was also easily applied to the mesh screen. The ratio used was 35:1, water: dry ingredients by weight. The slurries had to be poured immediately after being blended as it was observed that the particles separated rather quickly. The time this took was tested by pouring a slurry into a clear

test tube and starting a timer. The timer was stopped once the particles had visibly risen to the top of the test tube or sunk to the bottom. Slurries were applied to the mesh screens, then were left to drip and air dry for around five days before being placed in an oven at 60 degrees C for 24 hr to dry the sample thoroughly. The drying oven used can be seen in Figure 2c. This method produced a sample free of any attached soil particles and is relatively consistent from one sample to the next. After the sample is dry, a roughly 28 by 30 cm sheet of the HM remained. Some examples of dried samples are shown in Figure 3.

The paper used came in large sheets. Because of this, it was first cut into small pieces to allow for more accurate weight measurements before being placed in a vessel and blended with a tackifier and water. The wood fibers arrived small, so no further work was done before adding them to the HM. Hemp hurds arrived as a mixture of small hemp hurd pieces and longer hemp fibers and were not modified before use. Guar gum and psyllium husk arrived as a very fine powder, so were added directly into the HM slurries, in contrast camelina meal arrived in flakes which were ground in a coffee grinder for approximately one min or until a particle size similar to that of the other tackifiers was reached (i.e., powder).

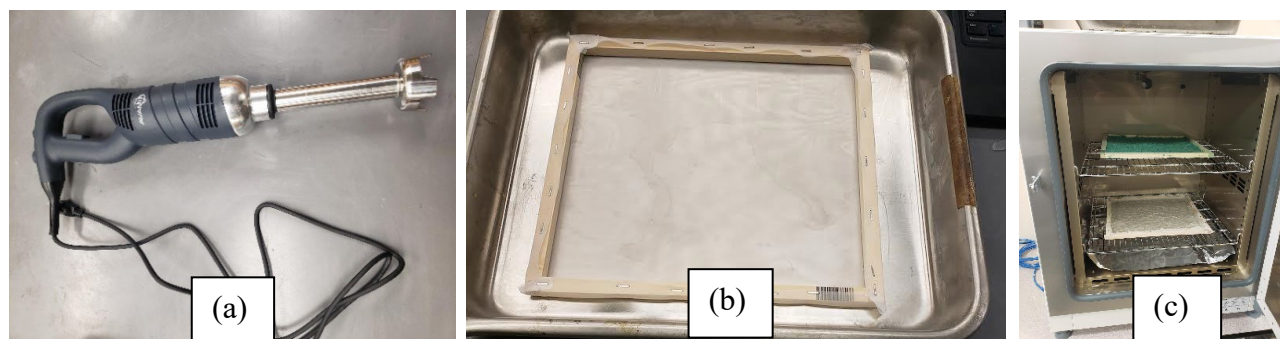


Figure 2 - Immersion blender for combining ingredients into slurry (a) nylon screen and frame used to create samples (b) drying oven used for drying containing paper and wood samples (c)

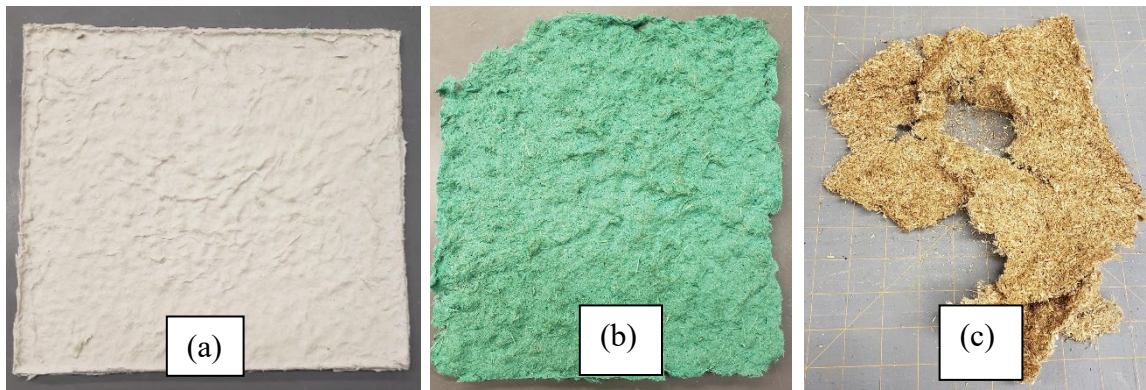


Figure 3 – Pictures of the 28 by 30 cm HM samples after drying and removing from nylon screen. The samples contain: paper (a), wood fiber (b), or hemp hurd (c) and 2% guar gum

3.2.2 Strength Testing

One of the most important properties of the HM is its ability to suppress weed growth. Inferences about weed suppression can be made based on HM's performance in strength testing. Both tensile strength and puncture resistance were tested. These tests helped determine which formulations would be more likely to hold up throughout the grow season and resist weed emergence (Verdú et al., 2020). The strength tests were done using a Mark-10 universal testing machine (Mark-10, Copiague, NY) equipped with 100 N load cell. For the tensile test, pieces of mulch were cut out of the sheets into 19 mm wide and 152 mm long strips. The strips were attached to the machine at each end using wedge grips. Tensile force was applied until rupture using cross-head speed of 1 mm/min as reported by Verdú et al. The setup is illustrated in Figure 4a. The force at rupture was recorded and used along with cross sectional areas at the break location to determine the tensile strength of each sample (Equation 1). The plot generated by the test sample can be seen in Figure 5 with the rupture force marked at the top of the curve. For the puncture test, first a fixture was designed. The fixture contained a hole for a probe to pass through while penetrating the sample. Clamps were used to hold the sample in place while it was

being punctured (Figure 4). The sheets were placed over the fixture and a 13.7 mm diameter probe was lowered down at a rate of 4 mm/min, as done by Verdú et al., onto the sheet until rupture, similarly to the tensile test which generated the same shaped plot as seen in Figure 5. Using the force at rupture, diameter of the probe, and the thickness of the sample at puncture location, a value for puncture resistance was obtained using Equation 2 below. Five replications were tested for both the tensile and puncture tests for each HM formulation. An example of a paper sample after both the puncture and tensile test can be seen in Figure 6.

It was hypothesized that that the paper would be stronger than the wood and hemp hurd samples based on the way they mixed in with water. The addition of tackifier and increase in tackifier concentration was expected to result in increases in both tensile strength and puncture resistance. Finally, it was hypothesized that the addition of larger hemp and wood fibers to the paper sample may help hold the samples together, increasing the overall strength. The paper broke down into a slurry that hardened as a solid material where the hemp and wood remained as larger fibers that were barely held together by tackier once dry.

$$\textit{Tensile Strength} = \frac{\textit{force at rupture}}{\textit{sample width at rupture} * \textit{sample thickness at rupture}} \quad [1]$$

$$\textit{Puncture Resistance} = \frac{\textit{force at rupture}}{\textit{probe diameter} * \pi * \textit{sample thickness at rupture}} \quad [2]$$

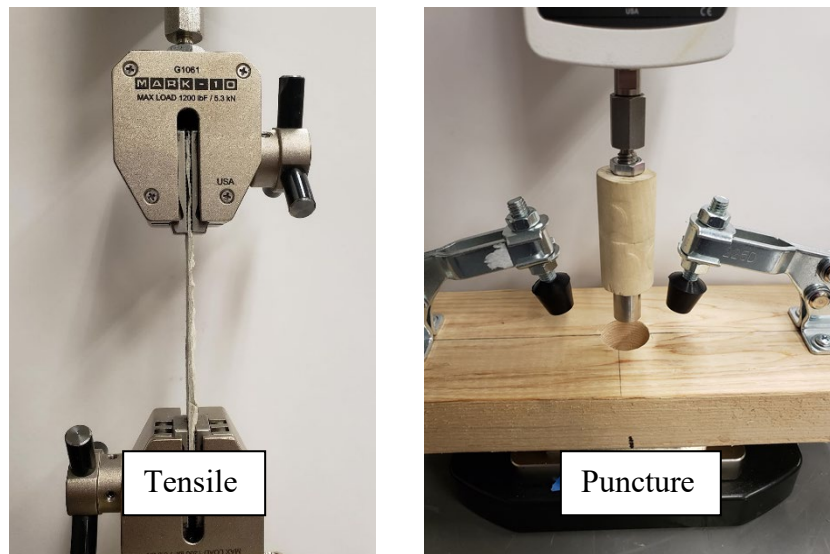


Figure 4 – Testing setups for tensile strength and puncture resistance tests

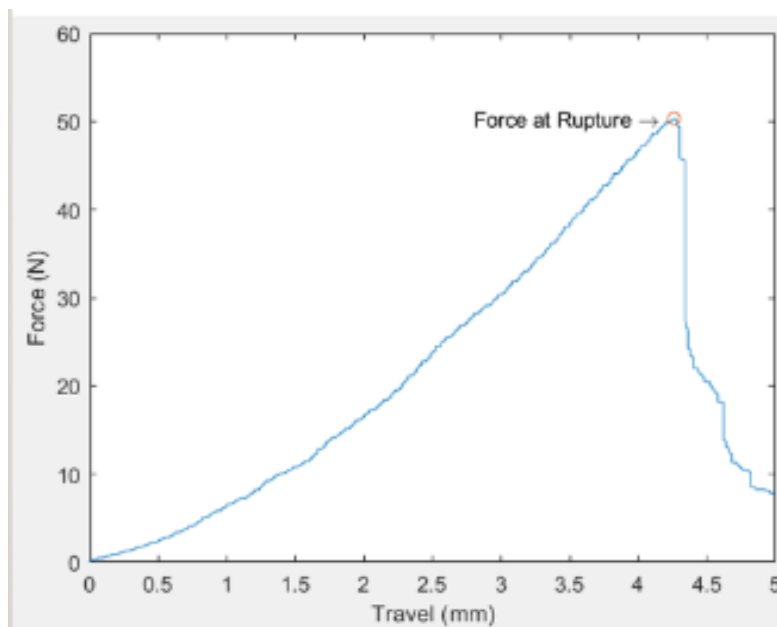


Figure 5 – Force versus travel plot to determine force at rupture

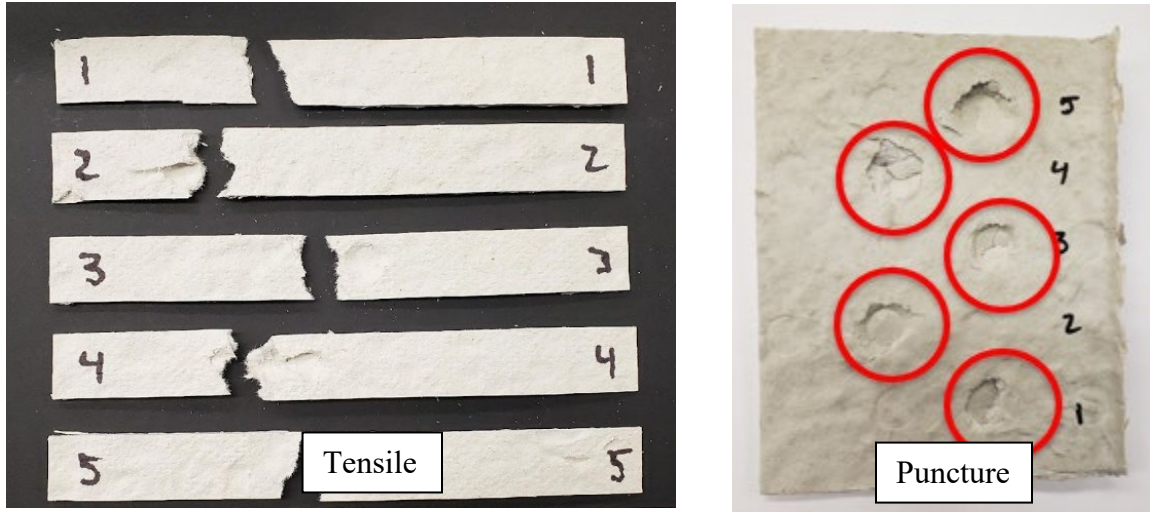


Figure 6 – Samples after completion of tensile and puncture resistance tests

3.2.3 Moisture Content Effects on Puncture Resistance

Because HMs are subjected to variable and often wet environment conditions, a test was conducted to see how HMs moisture content affects puncture resistance. This was done comparing guar gum to camelina meal as tackifiers with paper as the fibrous material. These were chosen because guar gum was the top performer in many tests and camelina meal was a potentially less expensive alternative. Each of P2GG, P2CM, P6GG, and P6CM were tested at four moisture levels, a control with 0% added moisture, 50%, 75%, or 100% moisture added by weight. The test was done by weighing the samples dry and then spraying the samples with a pressurized mister (Figure 7) until the target moisture levels were achieved. These samples were then all puncture tested while wet using the same procedure as above and repeated five times. These results were compared to the control samples to determine how moisture content affected the puncture resistance of the HM samples. It was hypothesized that since camelina meal is an oily tackifier it would handle the moisture better than the starchy material, guar gum.



Figure 7 – Pump sprayer that was used to add moisture to sample to test moisture content effects on puncture resistance

3.2.4 Rain Fastness

To provide all the benefits of a mulch the HM must stay intact over the course of a growing season. If the HM is washed away easily by rain, weeds may emerge defeating the purpose of the mulch. A rain fastness test was designed to compare how the different formulations break down during a rain event and how much material is washed away. For the rain fastness test, a device was designed to simulate rainfall at a rate of 1.52 cm/15 min. 1.52 cm of rainfall was used to match that used by Vaughn et al. (2012). The device consisted of three parallel 61 cm sections of 1.27 cm PVC pipe spaced 8.9 cm apart. Along the bottom of these pipes were 0.7 mm diameter holes spaced one in. apart. The samples were placed at an angle of 10 degrees to allow water and material to run off. After 15 min of exposure the samples were redried, and the initial and final weights were compared (final weight/initial weight) to determine rain fastness index (RFI). Figure 8 shows the rain fastness apparatus in use with both a paper and wood-based HM sample. The paper was expected to be the best fibrous material due to how well the fibers blend and adhere to one another. Camelina meal was expected to be the best tackifier at repelling water due to its high oil content and the results of previous studies.

To rule out the possibility of the exposure time of 15 min impacting the results, a test was done to compare the RFI with exposure times of 15 and 30 min. Also, to determine if the angle at which the sample is placed impacts the RFI, a test was done at 10 and 25 degrees from the ground. These were each done twice, and averages were compared. Another test was done where a sample went through the test, was then dried, and then went through another rain test.

After the samples were dried and weighed, they were tested for their puncture resistance. It was hypothesized that since the rain event resulted in material loss, the strength of the sample would also decrease. The puncture test procedure used was the same as before and was repeated five times for each formulation



Figure 8 – Paper and wood sample after one min of a 15 min rain fastness test

3.2.5 Density Variation

Another mechanical property evaluated was the area density of the different formulations. Measurements were taken to quantify volumetric densities amongst the different fiber types. Ten thickness measurements were taken and averaged for five different formulations along with length and width, allowing for a volume and therefore density value to be calculated. These formulations included a paper control (no tackifier), P6GG, 75PH6GG, 75PW6GG, and W6GG.

Due to the inaccuracy of this method and how time consuming it was it was not done for all formulations. It was observed that some formulations were smooth and uniform when making the samples, while others had variable amounts of material and clumps, despite being poured the same way. This variation in application rate could result in areas with variable amounts of HM, e.g., some areas being covered in too much HM and some areas not containing enough causing inconsistent HM thickness. Ideally, there would be no variation and the entire plot would contain the same amount of HM. Due to the samples' uneven, or bumpy surface, a thickness measurement was not able to be made accurately, which is needed for the denominator (volume) of the density calculation (mass/volume). Instead, the thickness was left out and the density was measured in grams per square meter (gsm). Since all the samples were made with the same application rate of 4483 Kg/ha (448 gsm), all the area density values should be the same. To quantify the clumping that was observed, the variance between five density measurements was calculated for each of the HM formulations. If the samples were perfectly uniform and all trials yielded the same values, the variance would be zero. Formulations with higher variance values meant larger differences in the five measured samples suggesting clumping and uneven application with those formulations. The results from this test were further analyzed to determine if a tackifier, or a fiber type caused higher or lower CV values. It was hypothesized that the paper/wood and paper/hemp blends would have the lowest variation because they were the most uniform formulations when making the samples.

3.2.6 Soil Adhesion

Another property important in ensuring the longevity of the HM is how well it adheres to the soil. The better the HM adheres, the less likely it is to be removed by wind or rain. A soil

adhesion test was performed that aimed to determine if some formulations adhered to the soil better than others. The soil used for this experiment was All-Natural Garden Soil (Kellogg Garden, Carson, CA). The soil used is regionally formulated and contains one or more of: (processed forest products, recycled forest products, arbor fines, peat humus, sphagnum peat moss) as well as composted poultry manure, and organic fertilizer. A tray was lined with 6 cm of soil (Figure 9a). Then a slurry was applied to the soil and left to dry (Figure 9b). It was removed and weighed when the sample was dry (Figure 9c). The weight was compared to the initial weight of dry ingredients to determine how much soil had adhered ($((\text{final weight} - \text{initial weight of dry ingredients}) / \text{area of HM} = \text{adhered soil per area})$). This test was repeated three times for each formulation and the average over the three trials was reported. The formulation that performed the best in the strength tests were expected to be the best in the soil adhesion test. This is because the strongest formulations have the most adhesion between fibers. It was assumed this adhesion would also happen between the HM and the soil resulting in more soil adhering.

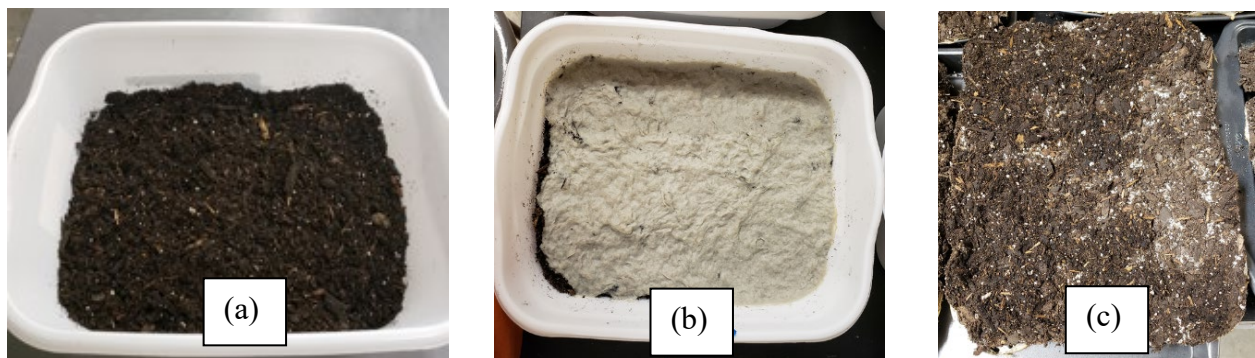


Figure 9 – 28 by 25 cm tub containing 6 cm of soil (a), tub of soil after slurry has been applied (b), HM sample removed with soil adhered (c)

3.2.7 Porosity

A very important mulch property is how well it can reduce soil evaporation, therefore, reducing the amount of water needed for the crop. This saves the farmer money and helps buffer the soil temperature from large fluctuations (O'Brien et al., 2018). To minimize soil evaporation as well as suppress weed emergence, a lower porosity HM is desired. Since porosity is “the degree of having minute spaces or holes through which liquid or air may pass” (“Porosity, n.”), it was quantified by the amount of water that the HM formulation could absorb in percent weight of original weight of the sample [(weight of sample at max water content - initial weight of sample)/initial weight of sample) * 100]. This was tested by first weighing a HM sample of size two by two in. (Figure 10a) and then submerging it in water for one min (Figure 10b). It was found experimentally that the HM samples would not absorb any more water after one min. The sample was then removed from the water, placed on a mesh screen (Figure 10c) to drip for one min, and weighed again to get the weight of the sample at max water content. This was repeated three times for each formulation. At this point in the project, it was clear the formulations containing just wood, just hemp, and 50PW were not going to qualify as top formulations. Because of this, just paper, 75PH, and 75PW were tested with all tackifiers at all concentrations. It was hypothesized that the HM formulations containing just paper would have a lower porosity than the blends as they were more solid and appeared to have fewer air pockets.

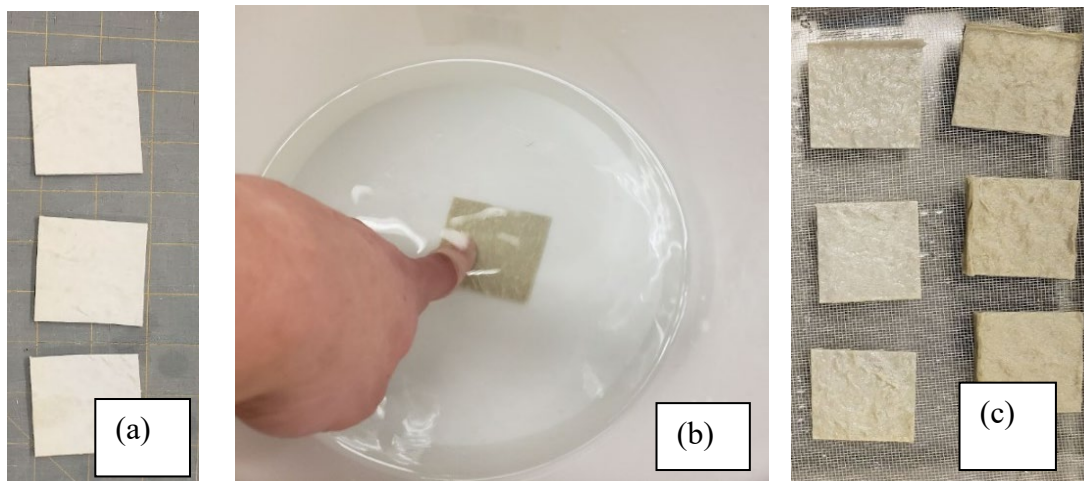


Figure 10 – Three stages of the max water absorption test. Dry samples (a), sample being submerged in water (b), excess water dripping off samples after submersion (c)

3.2.8 C:N Ratio

Crop yields and nutrient uptake depend on the ratio of carbon to nitrogen in the soil (C:N ratio). Higher crop yields have been observed when lower C:N ratio mulches were applied vs higher C:N ratio mulches (Truong et al., 2019). These effects are not seen during the cropping season but instead in subsequent seasons when the HM has biodegraded into the soil.

Decomposition of HM containing C and N can have short and long-term effects on available N in soil (Hadas et al., 2004) due to the high carbon content causing microbes to ‘tie up’ N.

Because of this, the C:N ratio of certain HM formulations was considered. The USDA-ARS located in Morris, MN tested the carbon and nitrogen contents of five formulations. These formulations were P6GG, P6PH, AND P6CM, 75PH6GG and 75PW6GG. These were selected because the paper was the top-performing material in most tests, so it was tested with all three tackifiers at the maximum concentration of 6%, while the paper and hemp blends were only tested with guar gum since it was the best-performing tackifier in most tests. It was expected that the carbon content would be similar amongst the five tested formulations. However, it was

unknown how the nitrogen content would compare, meaning the expected C:N ratios were unknown.

3.2.9 Determining top Formulations

The top formulations from each test were ranked and assigned point values based on performance ranging from one to ten. These point values were then put into a matrix where the different properties could be given different weights based on the importance of the property. The total points including the weighted adjustments, were then summed for each formulation allowing the point totals to be used to represent how well each formulation performed overall.

3.2.10 Statistical Model

To analyze the results of each test, statistical testing was performed in Minitab. An ANOVA was performed on the data with alpha value of 0.05 to determine if at least one group differs from the other groups. The assumptions for ANOVA are that the responses for each treatment have normal population distribution, the distributions have equal variance, and the data are independent. For the ANOVA test the null hypothesis was that means were equal for all formulations and the alternative hypothesis was that at least two means differed. If the results of the ANOVA test were significant, Fisher's least significant difference (LSD) method was used to determine differences between each group's mean value. Fisher's LSD procedure is a process for pairwise comparisons of several treatment groups (Meier, 2006). The main purpose of the Fisher's test is to compute the smallest significant difference between two means and denote significance when the difference is larger than the LSD (Williams et al., 2010). This test was used due to its low chances of Type II error. Results gave significant groupings denoted by

superscripts attached to data allowing for differences in means to be seen between the difference formulations. Results of the statistical testing can be found throughout the results and discussion section as well and additional data can be found in the Appendix.

RESULTS AND DISCUSSION

4.1 Blending of Fibers and Tackifiers

Preliminary blending trials were conducted to identify potential fiber and tackifier combinations. Both fibers and tackifiers were blended to determine if the combination of multiple materials enhances the mechanical properties. The results demonstrated some fiber materials were not promising on their own. This was first discovered with hemp hurds as the hemp hurd samples, regardless of the tackifier and tackifier concentration, were so crumbly that they could not be cut into the appropriate size pieces to be tensile, or puncture tested. The consistency resembled that of a graham cracker crust and crumbled into many pieces when touched as seen in Figure 3. If the sample could not be touched without breaking hours after drying, it is unlikely that the sample would ever successfully suppress weeds or survive the length of a growing season. The samples containing wood fiber were less fragile and could be tested in some of the tests. However, in the strength tests, formulations containing wood fiber as HM, had the lowest values of all formulations tested. Because of this, hemp hurds and wood fibers were blended with paper in hopes that the resulting HM would possess improved properties versus the different fibrous materials on their own.

Testing the formulations containing blended tackifiers showed some interesting results. The combination of the tackifiers had a negative effect on the strengths of the HM which can be seen in Table 2 and 3. This implies it would be better to lower the amount of guar gum used than to mix it with a cheaper tackifier such as camelina meal. Table 2 shows that even though the sample containing paper, 4.5% guar gum and 1.5% camelina meal (P4.5/1.5GG/CM) contained more guar gum (4.5% vs. 4%) and overall, more tackifier (6% vs. 4%) than the P4GG, it had less

than half the strength (0.58 MPa vs. 1.11 MPa). P2GG was also stronger with one third as much tackifier (2% vs 6%). This could also be seen in the case of paper and 3% of each guar gum and camelina meal (P3/3GG/CM) where, there was more guar gum and overall, more tackifier than with P2GG, yet the strength was significantly lower. This same interaction can be observed in Table 2 with the combination of psyllium husk and camelina meal.

Table 2 – Effects of camelina and guar on puncture strength (MPa). One-way ANOVA using Fisher LSD comparison 95% confidence.

Formulation	P6GG	P4GG	P2GG	P4.5/1.5 GG/CM	P3/3 GG/CM
Puncture Resistance	1.43 ^a	1.11 ^b	0.87 ^c	0.54 ^d	0.58 ^d

Values that share a superscript are not statistically different

Table 3 – Effects of camelina and psyllium on puncture strength (MPa). One-way ANOVA using Fisher LSD comparison 95% confidence.

Formulation	P6PH	P4PH	P2PH	P4.5/1.5 PH/CM	P3/3 PH/CM
Puncture Resistance	1.32 ^a	1.00 ^b	1.00 ^b	0.68 ^c	0.56 ^c

Values that share a superscript are not statistically different

4.2 Preparation of Samples

The HM manufacturing process developed at Montana State University was successful in producing HM samples with consistent results. The samples demonstrated uniform properties within the different formulations. The sections cut from various locations within the samples as well as from other replicate sheets showed similar densities, thicknesses, and material properties.

One concern was that too much material would be lost through the screen during the making of the samples causing a deviation from the desired application rate of 4483 Kg/ha. To

verify this, samples were weighed and compared to the weight of the dry ingredients used to make the sample and it was found that the samples lost only about 2% of material while drying. This loss could have been reduced by placing the slurry in a container that cannot drain through the bottom allowing all the water to leave the sample through evaporation. The 2% loss in weight was acceptable since it significantly reduced the sample preparation time. This method did not drastically change the application rate, and closely represented real time application to soil.

The estimated time it took for fibrous materials to separate was observed to occur rather quickly (Table 4). Meaning when such materials are used in the field a constant agitation will be required in the application system.

Table 4 - Time for particles to separate from slurry

Material	Paper	Wood	Hemp
time to separation	10 min	1 min	5 min

4.3 Strength Testing

The two strength tests, tensile and puncture resistance, produced similar results. Although the values were not the same, the trends in data were, for example, paper was much better than the other fibrous materials in both tests.

One of the main goals of HM is to suppress weed growth. Puncture resistance is the mechanical property used to measure the formulation's ability to suppress weed growth. These values were compared to the maximum puncture resistance value at which a weed may emerge, 0.5 MPa (Verdú et al., 2020). When looking at the puncture resistance results (Table 5) many paper formulations exceed this value. However, these formulations were made in a lab setting

and had not begun degradation or been exposed to the elements (especially precipitation) like they would be in the open field.

The results for the puncture resistance test ranged from 0.03 MPa to 1.43 MPa. These results are in agreement with those found by Verdú et al. who observed values ranging from 0.4 to 1.4 MPa with a max of 1.47 MPa for a paper-based HM containing wheat straw, and gypsum. These values fall short of the LDPE value of 3.87 MPa but the formulations on the upper end of the spectrum exceed 0.5 MPa.

The best performing formulations for the puncture resistance test all contained paper and these formulations contained either guar gum or psyllium husk as a tackifier. The best two formulations contained the maximum amount of tackifier at 6%. This suggests that guar gum and psyllium husk results in a stronger HM and an increase in tackifier concentration results in an increase in the puncture resistance. In addition, all formulations containing paper and a tackifier performed better than the 100% paper control with no tackifier, suggesting that the introduction of tackifiers increased the puncture resistance of the HM samples.

The tensile strength results (Table 6) have a larger range of values than the puncture resistance ranging from 0.01 MPa up to 3.22 MPa. The tensile strength test results are similar to the puncture resistance results in terms of best performing formulations. This was expected as both tests are testing the strength of the material. Once again, the best formulations were those containing paper and a tackifier. Except for 2% psyllium husk, 6% camelina meal and 2% camelina meal, the addition of tackifier to the paper increased the strength compared to the control sample that had no tackifier. The best tackifiers were once again guar gum and psyllium husk. These puncture resistance values were higher than those found by Calaramunt et al. (2020),

who observed a maximum tensile strength of 0.11 MPa. However, the HM formulations used in this study were different. They contained paper, wheat straw, rice hulls, and mushroom substrate. Still, the top performing formulation was that containing paper similar to our results.

The blends containing paper and either wood or hemp hurds did not perform as well as the paper on its own. This was most likely due to the way the paper separated or settled in the blended formulations. Rather than enforcing the paper like expected, the addition of wood and hemp hurd fibers disrupted the paper creating voids and air pockets within the samples. This decreased the force at rupture as well as increased the cross-sectional areas of the samples, both of which decreased the puncture resistance and tensile strength of the formulations. The 75PW6GG and 75PW4GG blends were not statistically different from the paper and camelina meal at 2% and 6%.

Table 5 - Mean puncture resistance (MPa) values for each formulation over 5 trials. One-way ANOVA using Fisher LSD comparison 95% confidence.

Formulation	Mean Puncture Resistance (MPa)	Formulation	Mean Puncture Resistance (MPa)
P6GG	1.43 ^a	75PW2PH	0.37 ^{lmno}
P6PH	1.32 ^a	75PH4GG	0.36 ^{mnp}
P4GG	1.11 ^b	75PW4PH	0.35 ^{mnpq}
P2PH	1.00 ^c	75PW2CM	0.34 ^{mnpqr}
P4PH	1.00 ^c	50PW6PH	0.34 ^{mnpqr}
P4CM	0.92 ^{cd}	50PW4PH	0.30 ^{nopqr}
P2GG	0.87 ^{de}	75PW4CM	0.30 ^{nopqr}
P6CM	0.85 ^{def}	50PW2GG	0.30 ^{nopqr}
P2CM	0.81 ^{ef}	50PW2PH	0.29 ^{opqr}
Paper	0.75 ^{fg}	50PW6GG	0.29 ^{opqr}
75PW6GG	0.66 ^{gh}	75PH4CM	0.28 ^{opqr}
75PW4GG	0.56 ^{hi}	75PH2CM	0.26 ^{pqr}
75PH2PH	0.52 ^{ij}	50PW4GG	0.25 ^{qr}
75PH6PH	0.49 ^{ijk}	75PH6CM	0.25 ^r
75PW2GG	0.47 ^{ijkl}	W4PH	0.07 ^s
75PW6PH	0.43 ^{klm}	W6GG	0.06 ^s
75PH4PH	0.43 ^{klm}	W6PH	0.05 ^s
75PH6GG	0.42 ^{klm}	W4GG	0.04 ^s
75PW6CM	0.41 ^{klm}	W2PH	0.04 ^s
75PH2GG	0.40 ^{klmn}	W2GG	0.03 ^s

Values that share a superscript are not statistically different

Table 6 - Mean Tensile strength (MPa) values for each formulation over 5 trials. One-way ANOVA using Fisher LSD comparison 95% confidence.

Formulation	Mean Tensile Strength (MPa)	Formulation	Mean Tensile Strength (MPa)
P6GG	3.22 ^a	75PW2PH	0.50 ^{klm}
P6PH	3.14 ^a	75PW2CM	0.45 ^{lmn}
P2GG	2.19 ^b	50PW6GG	0.44 ^{lmn}
P4GG	2.00 ^c	50PW6PH	0.43 ^{lmn}
P4PH	1.30 ^d	75PH6GG	0.41 ^{mno}
P4CM	1.28 ^d	50PW2PH	0.40 ^{mno}
P2PH	1.20 ^{de}	50PW4GG	0.37 ^{mno}
Paper	1.14 ^{de}	50PW4PH	0.37 ^{mno}
P6CM	1.10 ^{ef}	50PW2GG	0.35 ^{mno}
75PW4GG	0.96 ^{fg}	75PH2CM	0.33 ^{mno}
75PW6GG	0.90 ^{gh}	75PW4CM	0.33 ^{mno}
P2CM	0.87 ^{ghi}	75PW6CM	0.32 ^{no}
75PH2PH	0.77 ^{hij}	75PH6CM	0.29 ^{no}
75PH4PH	0.75 ^{hij}	75PH4CM	0.24 ^o
75PH6PH	0.74 ^{hij}	W4GG	0.03 ^p
75PW6PH	0.71 ^{ij}	W6PH	0.02 ^p
75PH4GG	0.69 ^j	W6GG	0.02 ^p
75PH2GG	0.66 ^{jk}	W4PH	0.02 ^p
75PW4PH	0.66 ^{jk}	W2GG	0.01 ^p
75PW2GG	0.60 ^{kl}	W2PH	0.01 ^p

Values that share a superscript are not statistically different

4.4 Moisture Content Effects on Puncture Resistance

It can be seen in Tables 7 and 8 that when moisture is added, even a small amount, the puncture resistance of the samples drops drastically. Contrary to the hypothesis, neither tackifier appears to handle the moisture better. This is something to consider as with only 50% moisture by weight the puncture resistance drops below 0.50 MPa in all formulations. The 0.5 MPa being the maximum puncture resistance value that a weed can penetrate. Considering that the HM takes hours to dry and maybe even days depending on weather, this creates opportunities for weeds to penetrate the mulch layer. Neither tackifier type nor tackifier concentration seemed to mitigate the moisture impacts meaning that the high oil content in camelina meal did not help the HM maintain strength when wet.

Table 7 - Comparison of puncture resistance (MPa) amongst 2% guar gum and camelina meal at different moisture levels. One-way ANOVA using Fisher LSD comparison 95% confidence.

Added moisture content by weight	0%	50%	75%	100%
P2GG	0.87 ^a	0.37 ^{bcd}	0.40 ^{bcd}	0.19 ^{de}
P2CM	0.81 ^a	0.44 ^{bc}	0.31 ^{cde}	0.29 ^e

Values that share a superscript are not statistically different

Table 8 - Comparison of puncture resistance (MPa) amongst 6% guar gum and camelina meal at different moisture levels. One-way ANOVA using Fisher LSD comparison 95% confidence.

Added moisture content by weight	0%	50%	75%	100%
P6GG	1.43 ^a	0.46 ^c	0.39 ^c	0.35 ^c
P6CM	0.85 ^b	0.17 ^d	0.20 ^d	0.20 ^d

Values that share a superscript are not statistically different

At the end of this project a new recycled paper source was introduced. It was combined with 2%, 4% and 6% guar gum and was tested for tensile strength and puncture resistance. These results were then compared to those gathered with the virgin U-Haul paper. These results can be seen in Tables 9 and 10. Table 9 shows that at all levels of tackifier, U-Haul paper was significantly greater in tensile strength. Table 10 shows that at 2% and 4% guar gum, the two types of paper did not significantly differ in puncture resistance while at 6% it did.

Table 9 – Comparison of tensile strength (MPa) of HM containing paper from different sources. One-way ANOVA using Fisher LSD comparison 95% confidence.

Tensile Strength	2% GG	4% GG	6% GG
U-Haul Paper	2.19 ^b	2.00 ^b	3.23 ^a
Recycled	0.90 ^d	1.21 ^c	1.45 ^c

Values that share a superscript are not statistically different

Table 10 – Comparison of puncture resistance (MPa) of HM containing paper from different sources. One-way ANOVA using Fisher LSD comparison 95% confidence.

Puncture Resistance	2% GG	4% GG	6% GG
U-Haul Paper	0.87 ^{cd}	1.11 ^b	1.43 ^a
Recycled	0.75 ^d	0.96 ^{bc}	0.93 ^{bcd}

Values that share a superscript are not statistically different

4.5 Rain Fastness

Rain fastness testing was first done with the paper formulations and two replications were done due to the length of time this test took. After these initial results were analyzed, it was observed there was little variance between replications. Due to the length of time required for

this test and the small difference in results, only one replication was done for the remaining formulations. The paper formulations retained more mass than the wood samples. This is consistent with the test done by Holt et al. (2005) that showed at a similar application rate, the wood samples lost the most material at 8%. The others lost around 5% with the best performing formulation being rice hulls with only 1.5% loss in weight. Additionally, tackifiers were compared to see if formulations containing camelina meal held up better to the rain test than the guar gum samples which was found in an experiment done by Vaughn et al. (2012) that looked at tackifier alternatives to guar gum. This was done by looking at average RFI values across the three tackifier types. There was no difference in RFI values across the three tackifiers (Table A.8, p-value = 0.588).

The rain fastness results were compared to a control sample containing just paper and no tackifier which had a RFI of 0.94 (Table 11). The control was among the highest values observed during the test suggesting that the addition of tackifiers may not decrease the amount of material lost during a rain event.

The test was done for 30 min and compared with the results of the 15 min test for P2GG. Although this doubled the amount of water that fell on the sample, it did not affect the RFI as they were both 0.93 suggesting most of the material loss occurs prior to the first 15 min. When the different angles were compared it was found that at 10 degrees, the RFI was 0.93 and was 0.95 when the angle was increased to 25 degrees from the ground. This was likely due to a smaller surface area exposed to water, as well as limiting the water that can pool up and soak into the sample. Finally, running a sample through two cycles of the rain fastness test yielded some interesting results. The RFI was 0.93 after the first cycle and 1.00 after the second meaning

material was only lost in the first cycle. This is consistent with the test that compared the exposure times which suggested the majority of the material lost is lost in less than 15 min.

Paper, 75PW, and 75PH were not significantly different from one another, but were better than both 50PW and wood as seen in Table 13. Also, the type of tackifier used did not affect the RFI.

When conducting the rain fastness test it was observed that while the paper remained rigid the entire time the wood formulations quickly returned to their slurry consistency. This allowed for the drops of water to easily penetrate the HM which can be seen in Figure 11. These holes did not go away after the sample dried leaving multiple potential paths for emerging weeds.

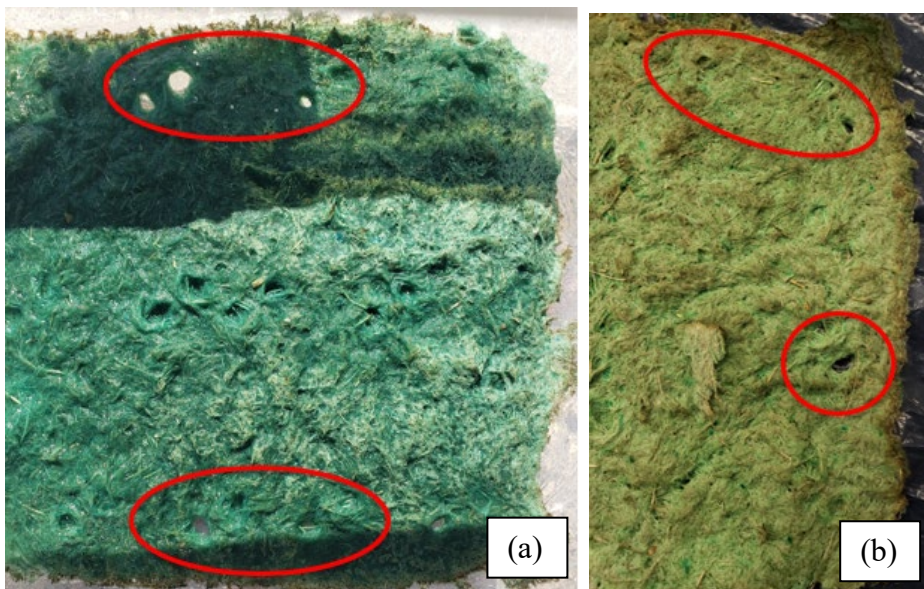


Figure 11 – Holes in wood sample due to rain fastness test while still wet (a) and when dry (b)

Table 11 - Results of rain fastness testing. * = average of 2 replications, one otherwise

Formulation	RFI	Formulation	RFI
P4CM	0.96*	75PH2CM	0.92
75PH4GG	0.95	75PH4CM	0.92
P4PH	0.94*	P4GG	0.91*
Paper	0.94	P6GG	0.91*
75PW2PH	0.94	75PW6GG	0.91
75PW4PH	0.94	50PW2CM	0.91
75PW2CM	0.94	50PW4GG	0.90
75PW4CM	0.94	50PW2GG	0.89
75PH2GG	0.94	50PW6GG	0.89
74PH6CM	0.94	50PW4PH	0.89
P2GG	0.93*	50PW6PH	0.89
P6PH	0.93*	50PW4CM	0.89
P2CM	0.93*	W6CM	0.89
75PW4GG	0.93	50PW2PH	0.88
75PW6PH	0.93	W2PH	0.88
75PH6GG	0.93	W4PH	0.88
75PH6PH	0.93	50PW6CM	0.87
P2PH	0.92*	W6GG	0.86
P6CM	0.92*	W6PH	0.85
75PW2GG	0.92	W2GG	0.82
75PW6CM	0.92	W2CM	0.82
75PH2PH	0.92	W4CM	0.80
75PH4PH	0.92	W4GG	0.76

Table 12 - RFI fiber type comparison. One-way ANOVA using Fisher LSD comparison 95% confidence.

Fiber Type	75PW	75PH	P	50PW	W
RFI	0.93 ^a	0.93 ^a	0.92 ^a	0.89 ^b	0.84 ^c

Values that share a superscript are not statistically different

After the samples underwent the rain fastness test, they were puncture tested to see how the rain simulation affected puncture resistance. The results are shown in Table 13. The paper formulations performed the best in the puncture resistance test. However, the paper formulations lost up to 35% of their original strength while the blends maintained their puncture resistance and, in some cases, this value increased by as much as 65%. Even though the blends appeared to have handled the rain simulation better with regards to puncture resistance, the puncture resistance of the paper formulations were still a lot higher. Puncture resistance values for the paper formulations ranged from 0.67 to 1.06 MPa while the blends ranged from 0.28 to 0.63 MPa with the exception of 75PW6GG which was 0.75 MPa. The increase of puncture resistance was due to the separation of the paper and the material it was blended with. This can be seen best with the 75PW samples since the wood fibers contain a green dye. Figure 12 shows the original sample which is the same light green color on both the top and bottom. However, after the rain fastness, it can be seen in Figure 12 that the paper and wood separated leaving the top white (paper) and the bottom green (wood). An animated cross section of the separation can be seen in Figure 13. This caused the paper to harden together without the interruption of wood fibers resulting in a sample with similar characteristics to the all paper samples. This separation only occurred during the rain fastness test and not while the samples were being made. This is likely due to the screens that were used to make the samples which removed most of the water very

quickly. The rain fastness test, however, fully saturated the samples with a constant supply of water for 15 min giving the fibers time to separate.

Table 13 - Average puncture resistance (MPa) after undergoing rain fastness test. One-way ANOVA using Fisher LSD comparison 95% confidence.

Formulation	Puncture Resistance (MPa)	Formulation	Puncture Resistance (MPa)
P4GG	1.06 ^a	75PH4PH	0.51 ^{ghijkl}
P6PH	0.93 ^{ab}	75PW2GG	0.50 ^{ghijkl}
P6GG	0.91 ^{ab}	75PW4CM	0.50 ^{hijkl}
P4CM	0.87 ^{abc}	75PH2GG	0.44 ^{ijklm}
P4PH	0.84 ^{bcd}	75PW6CM	0.43 ^{ijklm}
P2CM	0.75 ^{bcd}	75PH6PH	0.43 ^{ijklm}
75PW6GG	0.75 ^{bcde}	75PW4GG	0.42 ^{ijklm}
P2GG	0.71 ^{bcdef}	75PW2CM	0.41 ^{ijklm}
P6CM	0.69 ^{cdefg}	75PH2PH	0.38 ^{ijklm}
P2PH	0.67 ^{defgh}	75PH6GG	0.37 ^{ijklm}
Paper	0.66 ^{defgh}	75PH6CM	0.36 ^{ijklm}
75PW6PH	0.63 ^{efghi}	75PH2CM	0.36 ^{klm}
75PW2PH	0.55 ^{fghij}	75PH4GG	0.33 ^{lm}
75PW4PH	0.55 ^{fghijk}	75PH4CM	0.28 ^m

Values that share a superscript are not statistically different

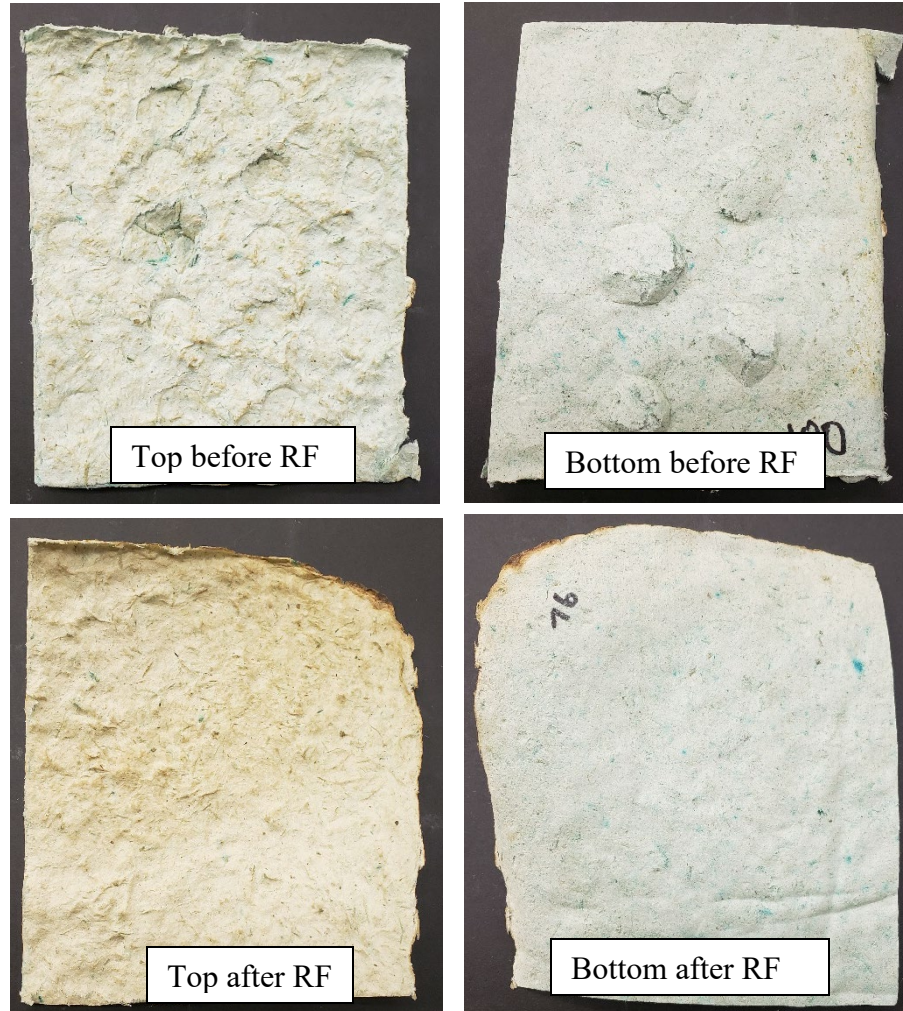


Figure 12 – 75PW sample before and after rain fastness test

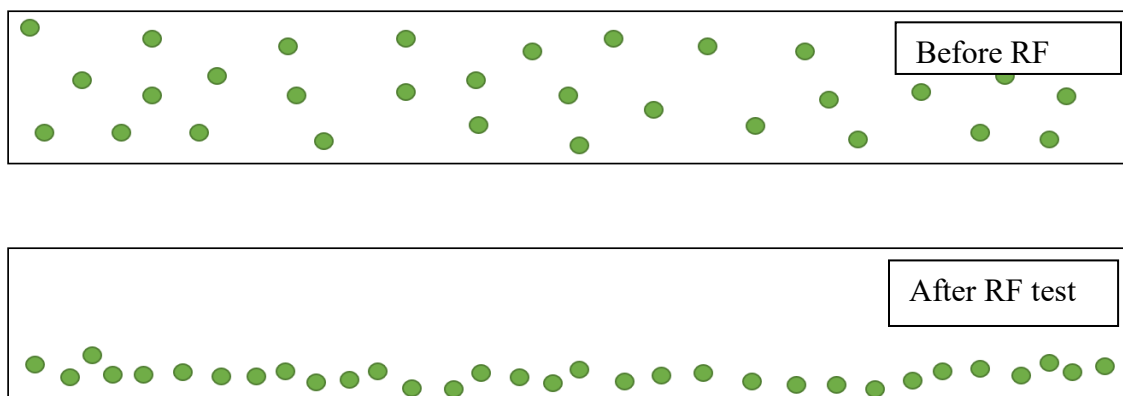


Figure 13 – Cross section of fiber separation after rain fastness test where green represents wood

4.6 Density Variation

Since an accurate thickness could not be gathered from the samples, a traditional mass/volume density calculation was only done for select formulations (Table 14). These results show that guar gum does not affect the density of HM as the paper control and P6GG have the same densities. Additionally, wood result in a much less dense HM and the addition of hemp and wood to paper appears to decrease the density of the HM. All formulations were measured in gsm and the coefficient of variation (C.V.) was looked at among the five replications for each formulation (Table 15). These C.V. values were then grouped into groups based on fiber type (Table 16). The coefficient of variation did not differ among the three tackifiers (Table A.11, p -value=0.884), suggesting that the tackifier may not affect the uniformity of the HM samples. However, significant density differences were observed amongst the different fiber types. Table 16 shows that 75PH and wood were not significantly different from one another but 75PH was significantly different than 75PW and paper suggesting that the fiber type affects the uniformity of the HM samples. This is due to the viscosity of the slurry. Because the same rate of water was used for all formulations, wood and hemp may be less absorbent than paper resulting in a less viscous fluid that spreads more uniformly and has less clumping. Additionally, the control sample containing just paper with no tackifier was slightly higher than other paper samples except for 2% and 4% camelina meal, suggesting the addition of tackifier may reduce the density variance resulting in a more uniform distribution of HM. These differences in viscosity and application rate can be fixed by adjusting the amount of water used to make the slurries. In the future it would be helpful to quantify the viscosities of the various slurries. This would allow consistent viscosities simplifying application. One method to do this would be using a device

like a paint viscosity measuring cup. This allows viscosities to be compared by how long they took to drain. One issue with this method is constant agitation would be needed to prevent the fibers from separating out of the slurry.

Table 14 – Density in g/cm³ for five HM formulations

Formulation	Density (g/cm ³)
Paper	0.184
P6GG	0.185
75PW6GG	0.128
75PH6GG	0.132
W6GG	0.070

Table 15 - Variation in gsm among five density measurements for each formulation reported in CV

Formulation	C.V.%	Formulation	C.V.%
75PH4PH	2.9	W2PH	13.4
W4GG	4.6	W6GG	13.5
75PH4CM	4.8	W6PH	13.9
75PW6CM	5.0	75PW4GG	14.0
75PH6CM	5.1	P2PH	14.1
W4PH	6.1	50PW4PH	14.5
75PW6PH	6.8	50PW2GG	15.4
75PH6GG	7.7	75PH2PH	15.6
P6GG	8.3	50PW4GG	15.7
75PH2GG	8.5	75PH4GG	16.5
75PH6PH	8.9	75PW2GG	19.2
P4PH	9.0	75PW2PH	19.2
75PH2CM	9.1	50PW6PH	21.2
75PW6GG	10.7	W6PH	21.2
W2GG	11.0	P2GG	21.7
P4GG	11.1	P6CM	21.8
50PW2PH	11.5	Paper	26.1
75PW2CM	12.1	P2CM	27.9
75PW4CM	12.2	75PW4PH	29.5
50PW6GG	13.1	P4CM	31.1

Table 16 - Mean C.V.% for each of the 4 fiber types. One-way ANOVA using Fisher LSD comparison 95% confidence.

Fiber	75PH	W	75PW	P
Mean C.V.%	8.8 ^a	11.6 ^{ab}	14.7 ^b	17.7 ^b

Values that share a superscript are not statistically different

4.7 Soil Adhesion

Soil adhesion results (Table 17) show that all formulations adhered to the soil with values ranging from 775 to 1875 gsm adhered soil. Formulations differed little with respect to soil adhesion due to large variances amongst replications. Formulations containing just wood or hemp and a tackifier were not tested because they were too fragile to peel off the soil once dried. Because of this only paper formulations and blends of paper and wood or hemp were used. The highest mean values observed were the paper formulations which aligns with the hypothesis that the strongest formulations would also have the best soil adhesion. However, soil adhesion values associated with various tackifiers were not higher than the paper control meaning the tackifier does not have a significant impact on how the HM adheres to the soil. Additionally, many of the 75PH blends did not differ from the paper formulations.

Table 17 - Average over 3 trials of soil adhered to each formulation reported in g/m². One-way ANOVA using Fisher LSD comparison 95% confidence.

Formulation	Adhered soil (g/m ²)	Formulation	Adhered soil (g/m ²)
Paper	1875 ^a	P6CM	1312 ^{cdefghij}
P2PH	1751 ^{ab}	75PH4GG	1287 ^{defghij}
P4PH	1705 ^{abc}	75PW4GG	1287 ^{defghij}
P2GG	1674 ^{abcd}	P4GG	1256 ^{efghij}
P6GG	1612 ^{abcdef}	75PH4PH	1194 ^{efghij}
75PH4CM	1581 ^{abcde}	75PW2GG	1194 ^{efghijk}
P6PH	1535 ^{abcdefg}	75PH2GG	1194 ^{efghijk}
75PH6CM	1519 ^{abcdefg}	75PW6PH	1178 ^{efghijk}
P2CM	1457 ^{bcdefg}	75PH2PH	1163 ^{fghijk}
75PH6PH	1426 ^{bcdefg}	75PW4CM	1132 ^{ghijk}
75PH2CM	1390 ^{bcdefgh}	75PW6CM	977 ^{hijk}
P4CM	1364 ^{bcdefgh}	75PW2PH	930 ^{ijk}
75PW6GG	1349 ^{bcdefghi}	75PW2CM	915 ^{jk}
75PH6GG	1349 ^{bcdefghi}	75PW4PH	7750 ^k

Values that share a superscript are not statistically different

4.8 Porosity

Porosity was assessed by measuring how much water a given amount of each HM formulation could absorb. Table 18 shows the results of the maximum absorption test. Values ranged from 433.7 - 987.9% increase from initial weight. Table 20 shows that, although the 75PW and 75PH blends did not differ from one another, they both differed from paper. These results were expected as the paper samples were much more dense and solid compared to other

formulations. The blends, however, were less dense and had a ‘spongy’ consistency. When the paper samples are made, the paper fibers hydrate better than the other fibrous materials creating a smooth pulp that hardens into a dense material with little room for air. Similar to what was discussed regarding strength testing, when the blends harden, the wood fibers, and hemp fibers do not hydrate and become a smooth slurry like the paper, causing air pockets and voids in the paper as the sample hardens. These air pockets are what causes the spongier feel, creating a more porous material, and resulting in higher maximum water absorption values. Also, the paper control had a higher maximum absorption than the paper formulations except for those containing camelina meal. This suggests that the addition of guar gum and psyllium husk creates a less porous HM whereas camelina meal has little to no effect on porosity.

Porosity also differed among tackifier types. Table 19 shows that although porosity of guar gum and psyllium husk formulations were not different from one another, they were both lower than the camelina meal formulations. These results suggest that samples containing camelina meal are more porous.

Table 18 - Average max water absorption for each formulation recorded as a percent increase from the initial weight of the dry sample. One-way ANOVA using Fisher LSD comparison 95% confidence.

Formulation	maximum water absorption	Formulation	maximum water absorption
75PH4CM	987.9 ^a	75PH2GG	723.3 ^{hi}
75PH6CM	942.1 ^b	75PH4GG	707.7 ^{ij}
75PH2CM	837.8 ^c	75PH4PH	671.5 ^{jk}
75PW2GG	835.0 ^c	75PH2PH	658.3 ^k
75PW6PH	821.3 ^{cd}	P6CM	603.9 ^l
75PW2CM	815.1 ^{cde}	P2CM	577.3 ^{lm}
75PW2PH	799.5 ^{cdef}	P4CM	572.2 ^{lm}
75PW4PH	789.7 ^{def}	Paper	567.3 ^{lm}
7PW6CM	780.8 ^{def}	P2GG	550.2 ^{mn}
75PW4CM	775.9 ^{efg}	P2PH	541.1 ^{mn}
75PW6GG	770.2 ^{fg}	P4GG	522.7 ⁿ
75PW4GG	761.9 ^{fgh}	P6PH	474.7 ^o
75PH6GG	739.3 ^{ghi}	P4PH	470.0 ^o
75PH6PH	726.6 ^{hi}	P6GG	433.7 ^o

Values that share a superscript are not statistically different

Table 19 - Comparison of max water absorption amongst the different tackifiers. One-way ANOVA using Fisher LSD comparison 95% confidence.

Tackifier	CM	GG	PH
Max Water Absorption	765.9 ^a	671.6 ^b	661.4 ^b

Values that share a superscript are not statistically different

Table 20 - Comparison of max water absorption amongst the different fiber types. One-way ANOVA using Fisher LSD comparison 95% confidence.

Fiber Type	75PW	75PH	Paper
Max Water Absorption	794.4 ^a	777.2 ^a	527.3 ^b

Values that share a superscript are not statistically different

4.9 C:N Ratio

Table 21 shows the results of the C:N ratio test. The carbon content of the five formulations is very similar, ranging from 47.5 to 48 except for 75PW6GG at 41.7. This was expected as all formulations are made up of mostly paper. However, the nitrogen content was higher in the P6GG and 75PW6GG resulting in a lower C:N ratio for the P6GG and 75PW6GG formulations. Psyllium husk and camelina meal contain less nitrogen than guar gum and 75PH6GG contains less nitrogen than 75PW6GG. The lowest C:N ratios were observed in 75PW6GG at 133:1 with values ranging from 133:1 to 364:1. A lower C:N ratio is better for crop yield (Truong et al., 2019), because when a ratio of 25:1 is exceeded, soil microbes will tie up N to achieve this ratio resulting in plant N deficiency (Booth et al., 2005). According to personal communication with a research soil scientist with the USDA-ARS, the ratios were all much higher than what is considered ideal for microbe and general fungi decomposition. Higher than ideal ratios were expected since cellulose-based mulch cannot have an ideal C:N ratio. This aligns with what was found by Zibilske et al. (2000) that increases in C in soil might be observed due to the breakdown of HM. This could be overcome in the future by large nitrogen additions to the HM.

Table 21 – Carbon and nitrogen content of five different HM formulations

Formulation	Carbon	Nitrogen	C:N Ratio
P6GG	47.5	0.335	142:1
P6PH	47.7	0.131	364:1
P6CM	47.7	0.183	261:1
75PH6GG	48	0.143	336:1
75PW6GG	41.7	0.314	133:1

4.10 Determining top Formulations

After each test, the top ten formulations for that test were given point values from ten to one. These point values were multiplied by a weight factor representative of the importance of the test. Puncture resistance before and after rain fastness were the highest weights at five followed by tensile strength at four. Porosity was given a three and density variation was given a two. Some tests were left out because the results were not significantly different across the many formulations. Rain fastness was not included as the results were quite similar across all formulations apart from the wood samples being lower. Soil adhesion was also not included because all formulations adhered to the soil and many formulations were not different. C:N ratio was also not included as only five formulations were tested and all ratios were higher than ideal.

The results of the top ten formulations can be seen in Table 22. The top formulation was P6GG. All nine of the paper formulations were among the top ten with the tenth being 75PW6GG.

Table 22 - Top performing formulations

1	P6GG	6	P2GG
2	P6PH	7	P2PH
3	P4GG	8	P6CM
4	P4PH	9	P2CM
5	P4CM	10	75PW6GG

CONCLUSION

In this project, 55 different HM formulations were tested for a variety of properties with the goal of finding the most promising formulations to move on to field trials. The hydromulch formulations contained fibrous materials such as paper, wood fiber, hemp hurds, or a combination of two. The fibrous material was mixed with a tackifier either guar gum, psyllium husk, or camelina meal at a concentration of 2%, 4% or 6% of the overall weight. The physico-mechanical tests conducted on these formulations included strength, rain fastness, density, soil adhesion, water absorption, and C:N ratio.

The tensile strength of various HM-formulations ranged from 3.22 MPa - 0.01 MPa. The puncture resistance varied from 1.43 MPa - 0.03 MPa. The strongest formulations were those made up of paper containing 6% of tackifiers and the weakest were those made up of wood fiber. The best performing tackifiers were guar gum followed by psyllium husk, which were also the two most expensive tackifiers. Furthermore, the two tackifiers were blended with camelina meal at various rates to lower the overall cost, hopefully without affecting the strength of the HM material. This resulted in a large drop in strength and was ruled out as an economical option. Blending paper with either wood or hemp hurds had similar results. The puncture test was repeated on select formulations at various moisture contents. The samples quickly lost strength as the water content increased regardless of the tackifier. The rain fastness test showed that tackifier type did not affect the amount of material lost in rain however the fibrous material did. The 100% paper (0.92), 75% paper/25% wood (0.93), and 75% paper/25% hemp (0.93) did not differ from one another but were better than 50% paper/50% wood (0.89) and 100% wood (0.84). After the rain fastness test, the paper samples lost between 4% and 37% of their original

puncture resistance while the puncture resistance increased with paper and wood and paper and hemp blends by as much as 65%. However, the paper formulations still had much higher puncture resistance values. The density test showed that type of tackifier had no impact on how uniformly the formulations spread but the fiber type did. It was observed that the coefficient of variation amongst sample densities were smaller in 75% paper/25% hemp formulations (8.8%) than 100% paper formulations (17.7%). Soil adhesion testing showed that all formulations adhered to the soil. Paper formulations had the highest mean values of adhered soil but were not significantly different from most of the other formulations. The porosity test found that the fiber type had a larger impact on the maximum absorption than the tackifier type did with 100% paper formulation being the least porous (527.3% max absorption) and 75% paper/25% hemp and 75% paper/25% wood being more porous (794.4% and 777.2% max absorption). The carbon and nitrogen content test of various formulations showed that guar gum results in a lower C:N ratio than the other tackifiers, and the paper and wood blend results in a lower ratio than the paper and hemp blend.

In terms of overall performance, the top five formulations were paper with 6% guar gum, paper with 6% psyllium husk, paper with 4% guar gum, paper with 4% psyllium husk and paper with 4% camelina meal (Table 22). All nine of the paper formulations were amongst the top ten and the only other formulation making the top ten was 75% paper/25% wood with 6% guar gum. Clearly, paper-based HM formulations were superior and may be suitable for organic farming as a biodegradable alternative to traditionally used LDPE.

The next steps in this project will be to test some of the top formulations in field trials with horticulture crops to further assess performance of HM formulations. These subsequent

experiments will help to understand how the different formulations degrade over the course of the growing season, how the degradation of the HM impacts the soils physical, chemical, and biological properties, how well they suppress weeds, and affect the crop yields. Additional lab work could include quantifying viscosities of the different formulations to ensure a consistent viscosity is used to simplify application. Also, work could be done to reduce the application rate and see how HM performance is affected. This could be a great way to reduce overall costs if similar performance could be achieved from less material.

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APPENDIX

STATISTICAL TESTING RESULTS

Table A.1 – ANOVA results for effects of guar and camelina on puncture resistance

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
form	4	2.6428	0.660703	70.83	0.000
Error	19	0.1772	0.009328		
Total	23	2.8200			

Means

formulation	N	Mean	StDev	95% CI
50/50 PH/CM	5	0.5640	0.0820	(0.4585, 0.6695)
75/25 PH/CM	5	0.6780	0.0936	(0.5725, 0.7835)
P2PH	5	1.0007	0.1534	(0.8952, 1.1062)
P4PH	5	0.9985	0.1157	(0.8930, 1.1041)
P6PH	5	1.3242	0.1076	(1.2187, 1.4297)

Pooled StDev = 0.113108

Table A.2 – ANOVA results for effects of psyllium and camelina on puncture resistance

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
formulation	4	1.8056	0.45140	35.28	0.000
Error	20	0.2559	0.01279		
Total	24	2.0615			

Means

formulation	N	Mean	StDev	95% CI
50/50 PH/CM	5	0.5640	0.0820	(0.4585, 0.6695)
75/25 PH/CM	5	0.6780	0.0936	(0.5725, 0.7835)
P2PH	5	1.0007	0.1534	(0.8952, 1.1062)
P4PH	5	0.9985	0.1157	(0.8930, 1.1041)
P6PH	5	1.3242	0.1076	(1.2187, 1.4297)

Pooled StDev = 0.113108

Table A.3 – ANOVA results for puncture resistance results

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Formulation	39	24.152	0.619293	86.39	0.000
Error	160	1.147	0.007168		
Total	199	25.299			

Means

Formulation	N	Mean	StDev	95% CI
50PW2GG	5	0.2992	0.0421	(0.2244, 0.3740)
50PW2PH	5	0.2936	0.0799	(0.2188, 0.3684)
50PW4GG	5	0.25480	0.01902	(0.18002, 0.32958)
50PW4PH	5	0.3040	0.0264	(0.2292, 0.3788)
50PW6GG	5	0.2870	0.0238	(0.2122, 0.3618)
50PW6PH	5	0.33740	0.02159	(0.26262, 0.41218)
74PW2GG	5	0.4716	0.0765	(0.3968, 0.5464)
74PW4GG	5	0.5576	0.0508	(0.4828, 0.6324)
74PW6GG	5	0.6568	0.1066	(0.5820, 0.7316)
75PH2CM	5	0.2634	0.0590	(0.1886, 0.3382)
75PH2GG	5	0.4018	0.0272	(0.3270, 0.4765)
75PH2PH	5	0.5180	0.1544	(0.4432, 0.5928)
75PH4CM	5	0.2846	0.1345	(0.2098, 0.3594)
75PH4GG	5	0.3616	0.1064	(0.2868, 0.4364)
75PH4PH	5	0.4294	0.0953	(0.3546, 0.5042)
75PH6CM	5	0.2464	0.0739	(0.1716, 0.3212)
75PH6GG	5	0.4168	0.0800	(0.3420, 0.4916)
75PH6PH	5	0.4916	0.0804	(0.4168, 0.5664)
75PW2CM	5	0.3436	0.0490	(0.2688, 0.4184)
75PW2PH	5	0.3714	0.0464	(0.2966, 0.4462)
75PW4CM	5	0.3008	0.0265	(0.2260, 0.3756)
75PW4PH	5	0.3526	0.0344	(0.2778, 0.4274)
75PW6CM	5	0.4146	0.0932	(0.3398, 0.4894)
75PW6PH	5	0.4344	0.1002	(0.3596, 0.5092)
P2CM	5	0.8118	0.1308	(0.7370, 0.8866)
P2GG	5	0.87340	0.01431	(0.79862, 0.94818)
P2PH	5	1.0007	0.1534	(0.9259, 1.0755)
P4CM	5	0.9210	0.1421	(0.8462, 0.9958)
P4GG	5	1.1114	0.0711	(1.0366, 1.1862)
P4PH	5	0.9985	0.1157	(0.9238, 1.0733)
P6CM	5	0.8465	0.1513	(0.7717, 0.9213)
P6GG	5	1.4298	0.1114	(1.3550, 1.5046)
P6PH	5	1.3242	0.1076	(1.2494, 1.3990)
Paper	5	0.7530	0.1382	(0.6782, 0.8278)
W2GG	5	0.03122	0.00998	(-0.04356, 0.10600)
W2PH	5	0.04010	0.01611	(-0.03468, 0.11488)
W4GG	5	0.04244	0.00653	(-0.03234, 0.11722)
W4PH	5	0.06814	0.01814	(-0.00664, 0.14292)
W6GG	5	0.06252	0.01917	(-0.01226, 0.13730)
W6PH	5	0.05206	0.01202	(-0.02272, 0.12684)

Pooled StDev = 0.0846651

Table A.4 – ANOVA results for tensile strength results

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
formulation	39	102.883	2.63802	130.90	0.000
Error	158	3.184	0.02015		
Total	197	106.067			

Means

formulation	N	Mean	StDev	95% CI
50PW2GG	5	0.3476	0.0555	(0.2222, 0.4730)
50PW2PH	5	0.4008	0.1014	(0.2754, 0.5262)
50PW4GG	5	0.3746	0.0686	(0.2492, 0.5000)
50PW4PH	5	0.3736	0.0801	(0.2482, 0.4990)
50PW6GG	5	0.4363	0.0415	(0.3109, 0.5617)
50PW6PH	5	0.4298	0.0393	(0.3044, 0.5552)
75PH2CM	5	0.3290	0.0554	(0.2036, 0.4544)
75PH2GG	5	0.6610	0.1300	(0.5357, 0.7864)
75PH2PH	5	0.766	0.317	(0.641, 0.891)
75PH4CM	5	0.2362	0.0691	(0.1108, 0.3616)
75PH4GG	5	0.6916	0.0887	(0.5662, 0.8170)
75PH4PH	5	0.7512	0.1673	(0.6258, 0.8766)
75PH6CM	5	0.2944	0.0507	(0.1690, 0.4198)
75PH6GG	5	0.4130	0.0602	(0.2876, 0.5384)
75PH6PH	5	0.7392	0.1760	(0.6138, 0.8646)
75PW2CM	5	0.4458	0.1062	(0.3204, 0.5712)
75PW2GG	5	0.5994	0.1325	(0.4740, 0.7247)
75PW2PH	5	0.504	0.347	(0.378, 0.629)
75PW4CM	5	0.3264	0.0898	(0.2010, 0.4518)
75PW4GG	5	0.9615	0.1897	(0.8361, 1.0869)
75PW4PH	5	0.6578	0.1395	(0.5324, 0.7832)
75PW6CM	5	0.3232	0.1130	(0.1978, 0.4486)
75PW6GG	5	0.8954	0.2157	(0.7700, 1.0208)
75PW6PH	5	0.7092	0.1906	(0.5838, 0.8346)
P2CM	5	0.8735	0.0904	(0.7481, 0.9989)
P2GG	5	2.190	0.258	(2.065, 2.315)
P2PH	5	1.1957	0.0847	(1.0703, 1.3211)
P4CM	5	1.2821	0.1805	(1.1567, 1.4075)
P4GG	5	1.9956	0.1211	(1.8703, 2.1210)
P4PH	5	1.303	0.226	(1.178, 1.429)
P6CM	5	1.1012	0.1950	(0.9758, 1.2266)
P6GG	4	3.2247	0.0753	(3.0845, 3.3649)
P6PH	5	3.1372	0.1204	(3.0118, 3.2626)
Paper	5	1.1414	0.1557	(1.0160, 1.2668)
W2GG	5	0.00878	0.00470	(-0.11661, 0.13418)
W2PH	5	0.007104	0.000556	(-0.118289, 0.132498)
W4GG	5	0.02819	0.00643	(-0.09720, 0.15359)
W4PH	5	0.01535	0.00254	(-0.11004, 0.14075)
W6GG	5	0.01790	0.00326	(-0.10749, 0.14330)
W6PH	4	0.02467	0.00490	(-0.11552, 0.16487)

Pooled StDev = 0.141962

Table A.5 – ANOVA results for puncture resistance at various moisture levels at 2% tackifier

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
form	7	0.7520	0.10742	8.88	0.000
Error	32	0.3873	0.01210		
Total	39	1.1392			

Means

form	N	Mean	StDev	95% CI
cp2cm	5	0.5016	0.0687	(0.4014, 0.6018)
cp2gg	5	0.6762	0.0896	(0.5760, 0.7764)
p2cm-100	5	0.2930	0.0959	(0.1928, 0.3932)
p2cm-50	5	0.4416	0.1369	(0.3414, 0.5418)
p2cm-75	5	0.3124	0.1152	(0.2122, 0.4126)
p2gg-100	5	0.1944	0.0802	(0.0942, 0.2946)
p2gg-50	5	0.3749	0.1613	(0.2747, 0.4751)
p2gg-75	5	0.3958	0.1020	(0.2956, 0.4960)

Pooled StDev = 0.110010

Table A.6 – ANOVA results for puncture resistance at various moisture levels at 6% tackifier

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
form2	7	6.5519	0.93599	85.10	0.000
Error	32	0.3520	0.01100		
Total	39	6.9039			

Means

form2	N	Mean	StDev	95% CI
cp6cm	5	0.8465	0.1513	(0.7509, 0.9420)
cp6cm-100	5	0.1960	0.0423	(0.1005, 0.2915)
cp6cm-50	5	0.1720	0.0565	(0.0765, 0.2675)
cp6cm-75	5	0.1960	0.0916	(0.1005, 0.2915)
cp6gg	5	1.4298	0.1114	(1.3343, 1.5253)
p6gg-100	5	0.3542	0.1141	(0.2587, 0.4497)
p6gg-50	5	0.4630	0.0716	(0.3675, 0.5585)
p6gg-75	5	0.3946	0.1455	(0.2991, 0.4901)

Pooled StDev = 0.104875

Table A.7 – ANOVA results for virgin vs recycled paper comparison

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
FORM PUNCTURE	5	1.4144	0.28288	12.51	0.000
Error	24	0.5429	0.02262		
Total	29	1.9573			

Means

FORM PUNCTURE	N	Mean	StDev	95% CI
NP2GG	5	0.7476	0.2151	(0.6088, 0.8864)
NP4GG	5	0.962	0.235	(0.823, 1.101)
NP6GG	5	0.9290	0.1294	(0.7902, 1.0678)
P2GG	5	0.87340	0.01431	(0.73458, 1.01222)
P4GG	5	1.1114	0.0711	(0.9726, 1.2502)
P6GG	5	1.4298	0.1114	(1.2910, 1.5686)

Pooled StDev = 0.150396

Table A.8 – ANOVA results for rain fastness tackifier comparison

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
TACK	2	0.001693	0.000846	0.54	0.588
Error	51	0.080339	0.001575		
Total	53	0.082031			

Table A.9 – ANOVA results for rain fastness fiber comparison

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
FIBER	4	0.05799	0.014497	29.54	0.000
Error	49	0.02404	0.000491		
Total	53	0.08203			

Means

FIBER	N	Mean	StDev	95% CI
50pw	9	0.89000	0.01118	(0.87516, 0.90484)
75PH	9	0.93000	0.01118	(0.91516, 0.94484)
75pw	9	0.93000	0.01118	(0.91516, 0.94484)
p	18	0.92444	0.01886	(0.91395, 0.93494)
w	9	0.8400	0.0433	(0.8252, 0.8548)

Pooled StDev = 0.0221518

Table A.10 – ANOVA results for puncture resistance after rain fastness

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
formulation	27	5.952	0.22044	9.21	0.000
Error	112	2.679	0.02392		
Total	139	8.631			

Means

formulation	N	Mean	StDev	95% CI
50PW2GG	5	0.3476	0.0555	(0.2367, 0.4584)
50PW2PH	5	0.4008	0.1014	(0.2900, 0.5116)
50PW4GG	5	0.3746	0.0686	(0.2638, 0.4854)
50PW4PH	5	0.3736	0.0801	(0.2628, 0.4844)
50PW6GG	5	0.4363	0.0415	(0.3255, 0.5471)
50PW6PH	5	0.4298	0.0393	(0.3190, 0.5406)
75PH2GG	5	0.6610	0.1300	(0.5502, 0.7719)
75PH4GG	5	0.6916	0.0887	(0.5807, 0.8024)
75PH6GG	5	0.8954	0.2157	(0.7845, 1.0062)
75PW2GG	5	0.5994	0.1325	(0.4885, 0.7102)
75PW4GG	5	0.9615	0.1897	(0.8507, 1.0723)
P2CM	5	0.8735	0.0904	(0.7627, 0.9843)
P2GG	5	2.190	0.258	(2.079, 2.301)
P2PH	5	1.1957	0.0847	(1.0848, 1.3065)
P4CM	5	1.2821	0.1805	(1.1713, 1.3930)
P4GG	5	1.9956	0.1211	(1.8848, 2.1065)
P4PH	5	1.303	0.226	(1.192, 1.414)
P6CM	5	1.1012	0.1950	(0.9904, 1.2120)
P6GG	4	3.2247	0.0753	(3.1008, 3.3486)
P6PH	5	3.1372	0.1204	(3.0264, 3.2480)
Paper	5	1.1414	0.1557	(1.0306, 1.2522)
W2GG	5	0.00878	0.00470	(-0.10204, 0.11960)
W2PH	5	0.007104	0.000556	(-0.103714, 0.117923)
W4GG	5	0.02819	0.00643	(-0.08263, 0.13901)
W4PH	5	0.01535	0.00254	(-0.09547, 0.12617)
W6GG	5	0.01790	0.00326	(-0.09292, 0.12872)
W6PH	4	0.02467	0.00490	(-0.09923, 0.14857)

Pooled StDev = 0.124986

Table A.11 – ANOVA results for C.V.'s of area densities based on tackifier

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
TACKIFIER	2	17.06	8.528	0.17	0.843
Error	36	1783.17	49.533		
Total	38	1800.23			

Table A.12 – ANOVA results for C.V.'s of area densities based on fiber

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
FIBER	3	396.7	132.23	3.30	0.032
Error	35	1403.6	40.10		
Total	38	1800.2			

Means

FIBER	N	Mean	StDev	95% CI
75PH	9	8.79	4.63	(4.50, 13.07)
75PW	15	14.67	5.99	(11.35, 17.99)
P	9	17.66	8.30	(13.37, 21.94)
W	6	11.63	5.98	(6.38, 16.88)

Pooled StDev = 6.33258

Table A.13 – ANOVA results for soil adhesion results

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
form	27	6386	236.51	3.25	0.000
Error	55	4003	72.78		
Total	82	10389			

Means

form	N	Mean	StDev	95% CI
75PH2CM	3	46.47	6.61	(36.60, 56.34)
75PH2GG	3	40.26	8.10	(30.39, 50.13)
75PH2PH	3	39.39	9.08	(29.52, 49.26)
75PH4CM	3	53.44	5.48	(43.57, 63.31)
75PH4GG	3	43.50	4.45	(33.63, 53.37)
75PH4PH	3	40.35	8.54	(30.48, 50.22)
75PH6CM	3	51.42	3.37	(41.55, 61.29)
75PH6GG	3	45.24	1.99	(35.37, 55.11)
75PH6PH	3	48.04	10.69	(38.17, 57.91)
75PW2CM	3	30.96	8.04	(21.09, 40.83)
75PW2GG	3	40.27	2.88	(30.40, 50.14)
75PW2PH	3	31.49	3.93	(21.62, 41.36)
75PW4CM	3	38.38	9.10	(28.51, 48.25)
75PW4GG	3	43.09	14.72	(33.22, 52.96)
75PW4PH	3	26.36	6.99	(16.49, 36.23)
75PW6CM	3	32.9	17.9	(23.0, 42.8)
75PW6GG	3	45.24	3.29	(35.37, 55.11)
75PW6PH	3	39.69	6.90	(29.82, 49.56)
P2CM	3	49.18	6.09	(39.31, 59.05)
P2GG	3	56.30	10.82	(46.43, 66.17)
P2PH	3	59.18	3.79	(49.31, 69.05)
P4CM	3	45.97	4.26	(36.10, 55.84)
P4GG	3	42.26	11.81	(32.39, 52.13)
P4PH	3	57.53	7.47	(47.66, 67.40)
P6CM	3	44.14	8.20	(34.27, 54.01)
P6GG	2	54.3	22.8	(42.3, 66.4)
P6PH	3	51.90	1.93	(42.03, 61.77)
paper	3	63.36	5.29	(53.49, 73.23)

Pooled StDev = 8.53107

Table A.14 – ANOVA results for porosity results

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
form	27	168.677	6.24730	102.20	0.000
Error	56	3.423	0.06113		
Total	83	172.100			

Means

form	N	Mean	StDev	95% CI
p2cm	3	5.773	0.182	(5.487, 6.059)
p2gg	3	5.5017	0.1007	(5.2157, 5.7876)
p2ph	3	5.4105	0.0533	(5.1245, 5.6964)
p4cm	3	5.7219	0.0903	(5.4360, 6.0079)
p4gg	3	5.227	0.284	(4.941, 5.513)
p4ph	3	4.7004	0.0488	(4.4145, 4.9864)
p6cm	3	6.039	0.373	(5.753, 6.325)
p6gg	3	4.3368	0.0694	(4.0509, 4.6228)
p6ph	3	4.747	0.179	(4.461, 5.033)
paper	3	5.6727	0.0505	(5.3867, 5.9586)
ph2cm	3	8.378	0.573	(8.092, 8.664)
ph2gg	3	7.2331	0.1011	(6.9472, 7.5191)
ph2ph	3	6.583	0.191	(6.298, 6.869)
ph4cm	3	9.879	0.228	(9.593, 10.165)
ph4gg	3	7.0773	0.1044	(6.7913, 7.3632)
ph4ph	3	6.7146	0.1390	(6.4287, 7.0006)
ph6cm	3	9.421	0.298	(9.135, 9.707)
ph6gg	3	7.3930	0.1115	(7.1071, 7.6790)
ph6ph	3	7.266	0.445	(6.980, 7.552)
pw2cm	3	8.151	0.265	(7.865, 8.436)
pw2gg	3	8.350	0.190	(8.064, 8.636)
pw2ph	3	7.995	0.225	(7.709, 8.280)
pw4cm	3	7.7590	0.0361	(7.4731, 8.0450)
pw4gg	3	7.6185	0.1703	(7.3326, 7.9045)
pw4ph	3	7.8974	0.1176	(7.6115, 8.1834)
pw6cm	3	7.808	0.218	(7.522, 8.094)
pw6gg	3	7.702	0.252	(7.416, 7.988)
pw6ph	3	8.213	0.573	(7.927, 8.499)

Pooled StDev = 0.247241

Table A.15 – ANOVA results for porosity based on tackifier

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
tack	2	17.93	8.963	4.69	0.012
Error	78	149.10	1.912		
Total	80	167.03			

Means

mat	N	Mean	StDev	95% CI
p	27	5.273	0.569	(4.978, 5.569)
ph	27	7.772	1.164	(7.476, 8.067)
pw	27	7.9437	0.3273	(7.6482, 8.2391)

Pooled StDev = 0.771200

Table A.16 – ANOVA results for porosity based on fiber

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
mat	2	120.64	60.3188	101.42	0.000
Error	78	46.39	0.5947		
Total	80	167.03			

Means

mat	N	Mean	StDev	95% CI
p	27	5.273	0.569	(4.978, 5.569)
ph	27	7.772	1.164	(7.476, 8.067)
pw	27	7.9437	0.3273	(7.6482, 8.2391)

Pooled StDev = 0.771200