SARE Final Grant Report FNE04-515
Horticultural Weed Barrier Mats from Dairy Manure – Phase 2

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Goals

The goal of the project is to refine the process for manufacturing self – fertilizing horticultural weed control mats out of dairy manure. The use of recycled paper fiber to add strength and flexibility to the manure based horticultural weed barrier mats will be investigated. It is clear that we cannot provide any cost effective physical or chemical treatments that can render the short - grass like fibers with enough strength to produce a durable sheet for field use. The bulk of the work at North Carolina State University centered on discovering a dewaxing process to apply to waste waxed cardboard boxes. Developing environmentally responsible, biodegradable, manure fiber based paper products that will substitute for polyethylene groundcover is the solution to a source reduction problem and to a nutrient management problem from livestock waste. As well if waxed cardboard can be effectively recycled and combined with manure fibers to produce the groundcover then another solid waste product (waxed cardboard) can be utilized.

Farm Profile

Freund's Farm, Inc. is a 230 cow rotationally grazed dairy with 650 acres of crops and pastures and a managed 200 acre woodlot. The dairy operates under the guidance of a Comprehensive Nutrient Management Plan and Integrated Pest Management. All manure is processed through a methane digester and separated. The liquid is stored in a lined lagoon. Liquids are draglined onto appropriate fields twice a year.

Participants

The project leader was Matthew Freund. Perry Gardner served as the local engineer. North Carolina State University was hired to refine and process the manure and investigate alternative complementary fibers.

Project Activities

Manure samples were collected over a series of months to analyze and account for changes in the substrate before paper processing would begin. NCSU began developing methodologies for removing wax from old waxed corrugated containers. Manure was dried and shipped to North Carolina for processing when the technology was developed to be able to use the old waxed corrugated containers. Significant issues developed when the cow bedding was changed to a different type of sawdust. The manure was much more variable and the sawdust aggregates caused significant weaknesses in the process.

Results

This section quotes for the report submitted by Med Byrd of North Carolina State University:

Summary

The goal of the project was to manufacture a sheet using 70 % composted manure fiber and 30 % old corrugated containers. Unlike the first phase of the project, an attempt was made to use wax-treated old corrugated containers rather than regular, recyclable containers, because of their significantly lower cost. However, at least 80 % of the wax must be removed, to avoid problems during sheet formation. Bench-scale trials demonstrated about 50 % of the wax could be removed by using high temperatures and an emulsifier (stearic acid) during pulping, followed by dewatering. Due to the time constraints of the project (the sheets had to be made in time for planting), a significant quantity of ground cover sheet was made using composted manure fiber and regular old corrugated containers.

Introduction

The overall goal of this project is to produce a weed control mat/ground cover sheet, using a combination of composted manure fiber (CMF) and some reinforcing fiber.

In Phase 1 of the project, NC State was able to produce a basic sheet, using 70 % CMF and 30 % fiber from repulped old corrugated containers (OCC). The CMF fiber was mechanically refined to convert the coarse grass-like fibers into basic papermaking fibers. The sheet tested well in field trials.

In the current phase (Phase 2), an attempt was made to reproduce the results of Phase 1, but using waxed old corrugated containers (WOCC) instead. Because of the deleterious effects of wax on recycled paper, WOCC is currently landfilled instead of collected for recycling. Therefore, it is a potentially inexpensive source of reinforcing fiber when compared to OCC.

In order for WOCC to be used for any papermaking process, most of the wax must be removed. NC State has estimated that at least 80 % of the wax must be removed before even a crude sheet, like a weed control mat, can be made without excessive fouling of the papermaking equipment.

NC State proposed that it might be possible to remove a large portion of the wax using the following process:

- traditional repulping, but at higher temperatures and/or times, to liberate the wax from the fibers
- addition of an emulsifying agent to the repulping step, to cause the wax to enter a stable mixture with the liquid phase
- dewatering after repulping, to remove the liquid phase and thus the wax

For the current work, it was decided to conduct a series of bench-scale trials, using stearic acid as an emulsifying agent. Temperature and time during the repulping step were to be varied over a practical range. The amount of wax remaining in the pulp after dewatering could then be determined by solvent extraction.

If successful conditions could be established, the final goal of the work was to produce pilot quantities of weed control mat (2000 linear feet or more), using the new fiber source.

Methods and Materials

I. Bench-Scale Trials to Remove Wax from WOCC

Experiment 1

The objective of the first trial was to experiment with the equipment and discover any experimental limitations that maybe experienced during the work. Saturated WOCC boxes were collected from a local grocer in Raleigh, NC, cut into one inch square pieces, and soaked overnight in water. The WOCC squares were then added to 2 liters of water at 40°C to make up 3 percent consistency pulp slurry (note: 3% consistency is a common consistency for repulping in the secondary fiber industry) after pulping in the Adirondack 450H hydropulper. To determine the amount of WOCC material to charge to the blender, the consistency was measured by weighing a known amount of board and placing it into a 105°C oven for 24 hours. By using the oven dried weight, the consistency was determined. With this information the 3 percent consistency was calculated on OD fiber weight. The wax content was assumed to be 30 percent for all saturated waxed samples. The hydropulper was then started and adjusted to 200 rpm for 10 minutes. A small amount of pulp was then removed to determine if the board had been completely repulped. This experiment was then repeated at 20, 30, and 60 minutes at both 200 and 420 rpm. After the experiments were completed on the saturated wax board, the same trials were repeated with double curtain coated waxed board. The percent wax for the curtain-coated board was assumed to be 13 percent on OD fiber. The curtain-coated board was used because the Adirondack hydropulper was unable to pulp the saturated WOCC. However, the curtain coated WOCC board had similar results using the same conditions.

Experiment 2

To find a procedure that could repulp the WOCC board, an industrial Waring Blender was used instead of the Adirondack 450H hydropulper. Both types of waxed board were again cut into 1 inch squares and placed in the blender to make a 2 liter slurry at 3 percent consistency after hydropulping. The same procedure was used to determine the consistency of the pulp, but the board was not pre-soaked prior to use. The experiments were then run on the medium speed setting and run for 10 and 20 minutes at both 40 and 55°C starting temperatures. The pulp was then analyzed visually to determine if the WOCC had been successfully repulped. After visually inspecting the pulp, it was determined that this method would be suitable for the trial parameters.

Final Experiment

Once a procedure was determined, trials varying time, temperature, concentration of emulsifier, and rotor speed were conducted. The slurry was centrifuged in the Ellis Drier Company 26 inch extractor (centrifuge) for 10 minutes and the pulp and filtrate were then weighed. The final consistency of the pulp removed from the extractor was determined using an IR-drier. The centrifuged pulp was also analyzed for wax content using a soxhlet extraction apparatus. Table 1 below describes the 16 different experiments that were performed.

Table 1: List of trial parameters

Run #	Time (minutes)	Speed	Initial Temp. (°C)	Concentration of Emulsifier
1	10	Medium	40	0.0 %
2	20	Medium	40	0.0 %
3	10	Medium	55	0.0 %
4	20	Medium	55	0.0 %
5	10	High	40	0.0 %
6	20	High	40	0.0 %
7	10	High	55	0.0%
8	20	High	55	0.0 %
9	10	Medium	40	0.5% Stearic Acid on OD Fiber
10	20	Medium	40	0.5% Stearic Acid on OD Fiber
11	10	Medium	55	0.5% Stearic Acid on OD Fiber
12	20	Medium	55	0.5% Stearic Acid on OD Fiber
13	10	High	40	0.5% Stearic Acid on OD Fiber
14	20	High	40	0.5% Stearic Acid on OD Fiber
15	10	High	55	0.5% Stearic Acid on OD Fiber
16	20	High	55	0.5% Stearic Acid on OD Fiber

Pulping

The board used for these experiments was International Paper double curtain-coated WOCC that was acquired from Dr. Richard Venditti. The moisture content of the WOCC material was determined, using the procedure described previously. To determine the amount of wax in the WOCC board, a sample was placed into a soxhlet extractor. With the known amount of wax and the consistency of the board, the proper amount of board could be added to 2 liters of water to produce a 3 percent consistency slurry. The blender was placed in warm water to reach the proper temperature; the water was adjusted to the proper temperature and added to the blender. The board was placed in the blender and the lid was sealed. The blender was started and a stopwatch started simultaneously.

To recover the pulp a strainer was placed into a 5-gallon bucket with three layers of cheesecloth over it. Once the appropriate amount time has passed the blender was stopped and the temperature was recorded. The slurry was then immediately poured into the cheesecloth. The cheesecloth was tied and squeezed manually into the bucket. The pulp and cheesecloth were then swiftly carried to centrifuge. The slurry was then centrifuged for 10 minutes. The consistency of the pulp was then determined by use of the IR (infra-red) dryer. The filtrate was collected from the centrifuge and was added to the bucket. The filtrate and the pulp were saved for further testing and visual analysis.

An emulsifier was also tested using the same procedure for each condition. Stearic acid was chosen as the emulsifying agent for its wide availability and low price. The stearic acid was charged at 0.5% on OD fiber. Stearic acid is a solid at room temperature and has a melting point of 69.3°C. To dissolve the emulsifier into solution, it was added to the blender with the board and water. The blender was sealed and started before the WOCC was added to the blender.

Soxhlet Extraction

To test the material for it wax content a soxhlet extraction method was used.

The standard method for "Rosin in paper and paperboard" (TAPPI T408) was followed.

II. Pilot-Scale Fiber Preparation and Papermaking

CMF was received from Freund's Farm in a moist form, in tote sacks.

Before mechanical refining, it was necessary to remove the non-fibrous portion of the material, which is mostly soil-like particulate. This removal was accomplished by mixing the CMF with water to a solids content of approximately 1 %, then passing the slurry across a slanting screen with a slot size of approximately 0.15 inches. The particulate passed through the screen, while the coarse fibers were retained in a catch tank. The combined fibrous portion was dewatered by draining into a fine-mesh screen box.

Refining was conducted on a Sunds Defibrator CD-300 pilot refiner, equipped with 12-inch grooved plates and a 250-HP motor rotating at 3000 rpm. A plate gap of 0.3-0.5 mm was used, which resulted in a significant input of refining energy. A small amount of dilution water was injected continuously into the refiner. The refined fiber was captured in a screen box and dewatered.

OCC was obtained from a local recycler and pulped up at 3 % consistency. It was lightly refined, using a single pass (20 KW over no-load) through a Bauer Twin-Flow Refiner.

Papermaking was performed on the NC State pilot machine. The machine is 16 inches wide at the forming zone, and it trims to approximately 11 inches width at the reel. The machine features five vacuum flat boxes, two dewatering presses, and a large steam cylinder drying section. The refined CMF and OCC fiber slurries were combined, diluted to approximately 1.5 % total solids, and fed to the machine.

The finished sheet, approximately 11 inches wide, was made into rolls of 2-3 feet diameter. The rolls were shipped to Freund's Farm.

Results and Discussion

Bench-Scale Trials

After visually inspecting the pulp from the Adirondack 450H hydropulper at all conditions, it was discovered the WOCC board could not be efficiently repulped. After both types of WOCC squares had been placed in the hydropulper for 60 minutes, at 420 rpm, and 40°C, the sample was removed inspected visually for clumps and unpulped material. It was discovered that even after 60 minutes in the Adirondack 450H hydropulper, only the corrugated medium was repulped. It was then decided to conduct another method of repulping that would induce more shear into the pulp slurry.

Two liters of water was then added to the Waring Blender with enough curtain coated WOCC material to achieve a 3 percent consistency slurry. After 10 minutes at an initial temperature of 40°C on the medium speed, the Waring Blender removed all of the clumps from the slurry and was determined to be successful. The time, speed, and temperature trials were then completed. After the slurry was centrifuged approximately 10 to 15 grams of the pulp was placed in the Soxhlet extractor to analyze it for wax content. The final wax content and trial parameters are displayed in Table 2 below.

Next, four pieces of 1-inch by 1-inch curtain coated board were placed in the soxhlet extractor to determine the original wax content. After 24 hours of extraction the unpulped board was removed, dried, and the wax content determined to be 13.6 percent. This wax content served as the basis to determine the wax removal efficiency and as a basis of comparison between samples.

From the experiments conducted, the trials when 0.5% stearic acid on OD fiber was added to the fiber slurry the process achieved the greatest wax removal efficiency. The stearic acid acted as an emulsifying agent that aided the wax into the liquid phase. Once in the liquid phase, the stearic acid-wax emulsion was removed during the centrifugation process. Figure 1 shows the effect stearic acid had on the wax removal efficiency.

The wax removal efficiency was calculated using equation 1.

Efficiency = (13.6 – Wax Content After Repulping) * 100 / 13.6 **Equation 1**

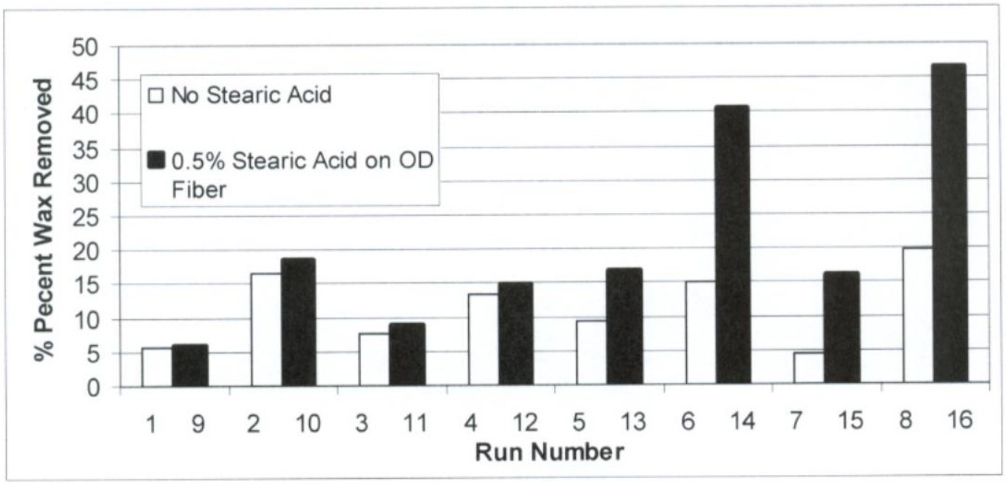
Holding all other parameters constant and adding stearic acid to the fiber slurry the experiments were able to achieve increased wax removal efficiencies. During the experiments the stearic acid was found to be very difficult to dissolve into the liquid phase. The reason for this problem is that stearic acid reacts quickly with the calcium in the hard tap water, preventing it from dissolving. Even though the stearic acid showed some positive results, they were very limited until the solution reached elevated temperatures. Runs 14 and 16 reached temperatures of 71 and 78 °C. At these temperatures the stearic acid melted (m.p. 69 °C) and entered the liquid phase and began to emulsify the wax particles. Once the stearic acid was melted into the liquid phase runs 14 and 16 achieved 40.68 and 46.60 percent wax removal efficiencies respectively.

Table 2: Results from the wax removal trials

Run #	Initial Temp. (°C)	Final Temp (°C)	Time	Speed	Emulsifier (% on OD)	Wax Content After Repulping (% on OD Fiber)
1	40	39	10	Medium	0	12.8220
2	40	48	20	Medium	0	11.3342
3	55	56	10	Medium	0	12.5729
4	55	62	20	Medium	0	11.4207
5	40	53	10	High	0	12.3139
6	40	70	20	High	0	11.5353
7	55	67	10	High	0	13.0035
8	55	79	20	High	0	10.9181
9	40	40	10	Medium	0.5	12.8082
10	40	47	20	Medium	0.5	11.0835
11	55	57	10	Medium	0.5	12.4006
12	55	63	20	Medium	0.5	11.5931
13	40	53	10	High	0.5	11.3221
14	40	71	20	High	0.5	8.06682
15	55	64.5	10	High	0.5	11.4054

16	55	78	20	High	0.5	7.2627

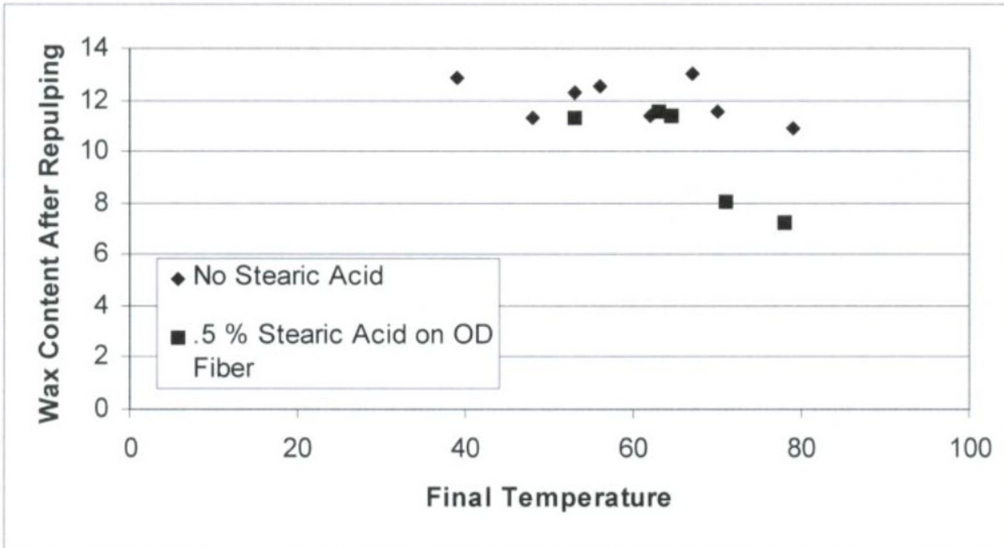
Figure 1: Condition comparison for runs varying 0.0% stearic acid and 0.5% stearic acid on OD fiber holding all other parameters constant.



To more efficiently remove wax it was discussed using an emulsifying agent with a lower melting point in the future.

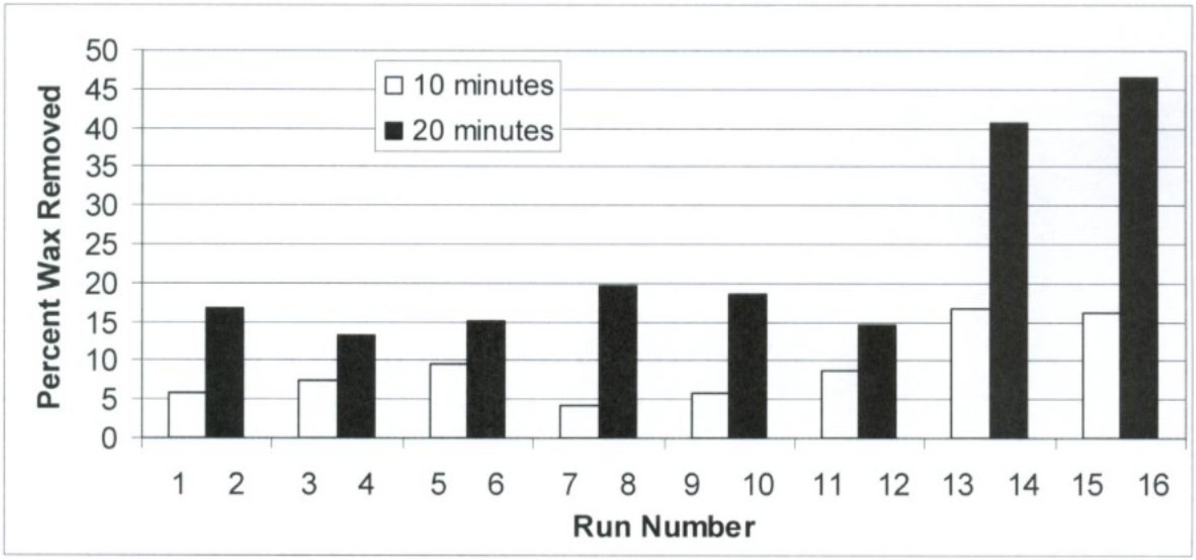
Figure 2 displays a graph of the final wax content versus the final temperature of the slurry. There was some mild effect of increased temperature on wax removal, but it was much milder than anticipated, and there was significant scatter in the data. The only effect temperature had on the process is that at temperatures above 70 °C the stearic acid was able to melt and emulsify the wax. The reason the temperature did not have a large affect is due to the fact that the melting point of the wax on the curtain-coated board was never reached. It was determined that the melting point of the wax was never reached because at the end of each trial unmelted wax particles were observed on the surface of the slurry.

Figure 2: Graph of Wax Content vs. Final Temperature



After further examination it was discovered that the wax removal efficiency depended much more on time instead of temperature. Holding all other variables constant the samples that were held in the Waring blender for 20 minutes achieved much higher wax removal efficiencies than did the samples pulped for 10 minutes. Figure 3 shows the effect of increased blending on the wax removal efficiency.

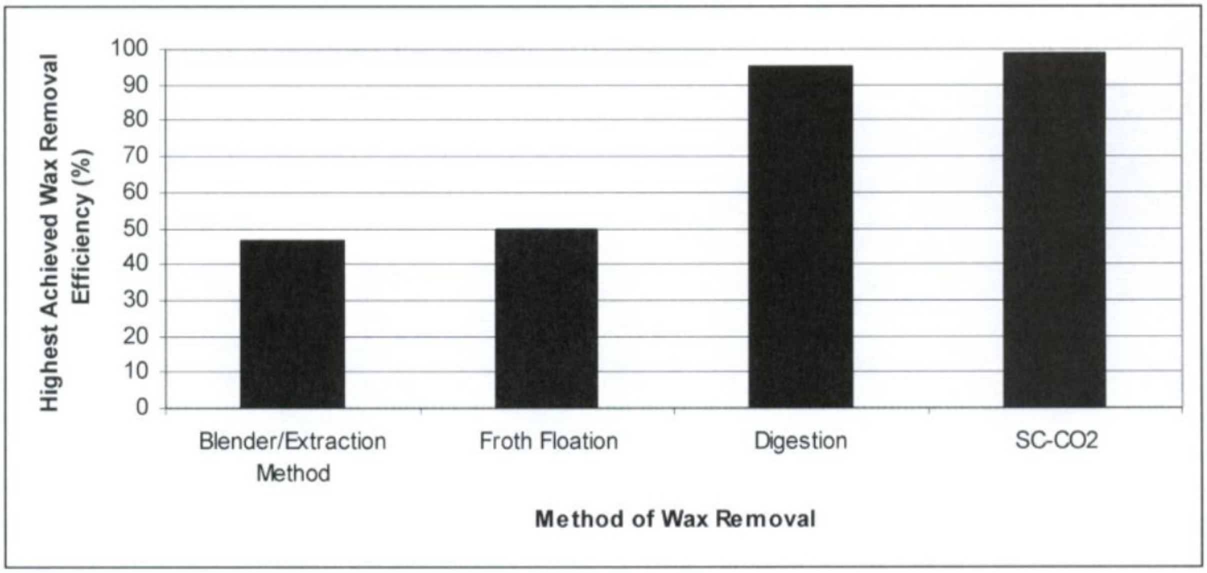
Figure 3: Effect of blending time on wax removal efficiency



From figure 3 one can see that the samples held in the blender for 20 minutes often had twice as much wax removed during centrifugation than did the samples that were blended for 10 minutes. The reason for this fact is most likely due to the increased wax-to-fiber bond breakage with the increased blending. The more times the blender hit the fibers the more the wax-to-fiber bonds were broken and the smaller the wax particulate material became. After analyzing the data it was discovered that the experiments conducted using the hydropulping/rapid dewatering method were reaching a limit of 50 percent wax removal efficiency, which is the same as noted in the literature for the froth flotation method. Most likely, the limiting factor was that many of the wax-to-fiber bonds were not broken, causing the wax to remain with the slurry. Thus, there is most likely a limit to the amount of the wax that can be removed or be detached from the fiber surface using mechanical means. In both the hydropulping/rapid dewatering and froth floatation methods used a hydropulper as the only means to break the wax-to-fiber bonds. Figure 4 below shows how the wax removal efficiency compares to previous experimental methods.

To achieve results greater than 50 percent wax removal, much more energy needs to be induced into slurry to break the wax-to-fiber bonds. From the literature, the digestion and super critical carbon dioxide methods both used increased temperature and pressure to achieve greater wax-to-fiber bond breakage and achieve wax removal efficiencies greater than 90 percent. However, there appears to be limit to amount of bond breakage that can be observed using only mechanical action.

Figure 4: Comparison of Wax Removal Methods



Additional trials were planned in an attempt to increase wax removal to at least 80 %. One means of accomplishing this goal was to use proprietary chemicals specifically developed to emulsify wax. However, due to the onset of planting season, it was necessary to move ahead with papermaking, using regular OCC.

Pilot-Scale Trials

It was estimated that 30-40 % of the original mass of the CMF material was lost during separation of fiber and particulate.

During refining, there was significant steam generation. Although the energy consumption was not measured, it will be significant, as is typical for a refiner mechanical pulping (RMP) process. The measured Canadian Standard Freeness of the pulp was 130.

Papermaking went fairly well, although the machine speed had to be limited to 30 feet/min (60-100 feet/min is normal) due to the poor drainage. There were few sheet breaks. There was evidence of a lot of sand and grit in the sheet, and it is recommended that a centrifugal cleaner may assist in the removal of these contaminants.

The final sheet was not very strong, but it was comparable to that made for Phase 1.

Conclusions

1. Using regular repulping methods, at high temperatures and stearic acid as an emulsifier, wax removal from WOCC was limited to approximately 50 %. This was far short of the 80 % minimum removal target required to use the WOCC material for papermaking.

2. With other variables fixed, increasing the repulping temperature increased wax removal.

3. With other variables fixed, increasing repulping time increased wax removal.

4. For all trials, the addition of stearic acid as an emulsifier increased wax removal.

5. It was possible to successfully produce a ground cover sheet using 70 % mechanically-refined CMF and 30 % unwaxed OCC.

Economics

It is too early to determine the economics of this technology. All work was done in a University setting on experimental equipment.

Assessment

The information gleaned and the paper produced need to be evaluated for scale up and field trials. We have glued the narrow sections together to mimic a 4 foot continuous plastic sheet. The sheets have been sent to Cornell University, USDA at Maryland, UCONN, and Freund's Farm Market for field evaluations. The next step will be to judge the effectiveness of the sheets to control weeds and durability over the growing season. If the sheets perform well then we need to assess the economics of making four foot wide sheets in a pilot plant or rented setting.

Adoption

The potential for this technology is enormous. We would be replacing a petroleum based product that requires significant removal and dumping costs with a biodegradable soil enhancement. The main impediment is proving the technology to justify the significant capital investment.

Outreach

Field trials are available for viewing at the above mentioned locations. Several tours have seen the paper placed at Freund's Farm Market. Digital photos are at the end of this report. Oral reports have been presented at the Canaan Valley Agricultural Cooperative Annual meeting and special summer meeting and at the Connecticut Department of Environmental Protection Concentrated Animal Feeding Operation Advisory Meeting.

Report summary

This project embodies the development of a potential symbiosis of the cattle and horticultural industries. Plastic waste volume would significantly lessen. Labor requirements for plastic clean up would be eliminated. Soil quality would improve. Animal waste nutrients would be applied where they are needed and away from where they are applied in excess.

Pictures: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