MATERIAL AND METHODS

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The experimental procedure pertaining to the study was conducted under the following heads.

3.1.	**PHASE I**	**:**	**Eliciting information and analyzing the selected cotton fiber waste**
3.2.	**PHASE II**	**:**	**Identifying possible products through pilot study**
3.3.	**PHASE III**	**:**	**Development of innovative sustainable products based on pilot studies using willow waste**
3.4.	**PHASE IV**	**:**	**Evaluation of the recycled products made from cotton waste**
3.5.	**Statistical Analysis**		

PHASE I

3.1. Eliciting information and analyzing the selected cotton fiber waste

There were few reviews published on the life cycle of spinning wastes. Hence, cotton mill owners and members of the cotton waste merchant association were approached to collect information regarding the types and uses of cotton wastes.

3.1.1. Examining the different types of spinning industry wastes

There are different types of spinning industry wastes, which are seen as a problem without any solution. The three most commonly found cotton wastes are: willow waste from Open End (OE) mills, un-saleable dirtied cotton bales stored in spinning mills and unprocessed cotton textile industry wastes.

3.1.2. Willow waste from OE mills

During cotton cultivation, it is inevitable to remove the kapas without seed and plant particles. Subsequently, in spinning, these trash particles are removed from cotton to obtain clean lint. The resultant spinning wastes are sent to the willow mill or the open end (OE) spinning mill for further cleaning of cotton fibers. The rack index in willow machine controls the cleaning level and approximately, 20-80% of cotton fibers are recovered in this process (Nityanand, 1984). The last trash from this process is called willow waste or willow dust (Plate 1). This is found in huge quantities and has not been much explored for an alternative end use.

3.1.2.1. Un-saleable cotton waste

Cotton bales from the field are usually stored in the godowns before going to the spinning mills for cleaning. Sudden rain or prolonged storage makes the cotton unmanageable. This stained and dirtied cotton waste releases foul odor, increases the risk of fire accidents and also leads to various lung diseases. This causes hindrance and is incinerated off (Plate 1).

3.1.2.2. Cotton processing industry wastes

Many cotton processing industries are tormented by storing huge cotton dusts without much recycling options. To understand this deeper, a leading denim exporting company was approached and samples were collected. The last scrap waste unutilized in the mills namely cotton dust, white poom and blue poom were selected (Plate 1). According to the data collected, the company produces six million meters of organic denim fabric per month. The processing exorcise 1, 02, 250 kgs of cotton dust, 58, 650 kgs of blue poom and about 7,850 kgs of white poom, (S. Kumar, Personal communication, December 8th, 2013).

Among the three wastes, there were very few researches on recycling willow waste. Hence the investigator selected it for further exploration.

3.1.3. Collection of information regarding the selected willow waste

The investigator interviewed professionals working with the willow waste industry to collect primary data about willow waste.

3.1.2.1. Conduct of interview

Interview schedule is a structured interview with a list of questions to test the assumption or hypothesis. Here the questions raised by the interviewer in face to face with interviewee serve as an effective means of collecting necessary information (Miller and Brewer, 2003). Since there were different cadre of people to collect information this method was chosen. The questions were framed to elicit information regarding the type of company, type of willow machine used, suppliers and customers, disease and infection caused by cotton dust, existing techniques in utilizing willow waste, cost, and seasonal based variation and pollution related awareness among them (Appendix 1).

3.1.2.2. Results of the interview

The interview revealed the fact that there were more than two hundred registered willow waste mills. Most of them were found to be small scales. Coimbatore and Madurai has the majority willow mills in South India. Willow waste was found to be purchased by the farmers, in and around Coimbatore, to supply it to their cattle: due to the high cost of cattle feed. Some use the water retention properties of this cotton waste, as compost bedding in their fields, especially in rice, coconut and turmeric farms. Majority of the willow machines were imported from John Green of London. The willow mill owners said that willow waste was utilized for paper making, bedding, upholsteries, socks making, low count bed sheets and cotton rope. The interviewees also pointed on the use of this cotton waste as a base for mushroom cultivation. Although there are few end uses locally found using the willow waste, it was reported, that the major quantity was thrown away to landfill without any end use. The analysis of facts collected during the interview also showcase that, apart from landfill pollution, it causes great threat for fire accidents and many breathing problems to the workers in willow mills. On the other hand, newer products utilizing this willow waste was found to be a very important recommendation to this crisis.

Based on the information collected it was understood that willow waste needs a solution to the disposal issue.

3.1.3. Identification, collection and analysis of the selected willow waste

Literature survey pointed out that every year approximately 30,000 tonnes of willow waste is expelled without any utilization (Suryawanshi, et al., 2013). Hence a research to explore this waste for innovative and sustainable products was momentous.

Five hundred kilograms of willow waste for Rs.3/ kg was purchased from Murugan willow mill, Ondipudur, Coimbatore (Tamilnadu, India). This was transported to Avinashilingam Institute for Home Science and Higher Education for Women, (Coimbatore) for trial studies.

3.1.4. Testing of willow waste for its composition

The pH, electrical conductivity, moisture, organic carbon, total nitrogen, total phosphorous, total potassium and C:N ratio, crude fiber, ash content, lignin, cellulose, calcium, magnesium, iron, copper, zinc, manganese, bacteria, fungi and actinomycetes, were tested at Environmental Sciences, Tamil Nadu Agriculture University, Coimbatore.

The test result shows that, willow waste has about 37% of lignin and 23% of cellulose, which makes it rich lignocellulosic waste. This can be an effective replacement for plastics or can be made into paper. The carbon nitrogen ratio of raw willow waste was 27.4, thus it can be biostimulated to be made into a compost and aid plant growth. With 47% of crude fiber, this willow waste can be made into nonwovens. As a source of cellulose, it can absorb and hold water. The lignin content acts as a natural binder to provide internal strength. Based on these insights, pilot studies were planned in Phase II.

PHASE II

3.2. Identifying possible products through pilot study

The prototypes and products from willow waste were designed based on the details on nature of waste, available quantity, ease in transportation of waste, preparation and logistics involved in real time application of the concept.

3.2.1. Pilot study for preparation of poly manure using willow waste with other textile wastes

Based on the previous work carried out by the investigator, it is clear that willow waste can be vermicomposted into effective bio manure. The study shows that raw willow waste has the recommended C/N ratio for composting. The resultant willow waste vermicompost was found to be a good source of carbon and other micronutrients. A sixty day pot culture study, confirmed that the prepared bio manure had positive implications on the growth parameters and plant products (Aishwariya, and Amsamani, 2012). Rather than using chemical additives to make the manure whole some, textile and agro-waste composts with specific nutrient can be profitable. According to statistical survey, eight million tonnes of wastes are eliminated annually from these, which reach the landfills. The report quotes that most of mill residues are non-toxic and when applied in low amounts to any vegetation helps in soil fertility. (Morris et al., 2000)

Hence a pilot study, exploring similar composts and combining it into a poly-manure for plants and crops was considered.

With this objective, sericulture waste, coir, jute and water hyacinth were collected and composted, individually (Plate 1). Sericulture waste was purchased from Ondipudur Silk Society (Coimbatore, India). It was mixed with cow dung in 1:4 ratio to control the foul odor and subjected to composting. In the same proportion, the jute from old sacks taken from domestic household was cut into one centimeter pieces and used. Water hyacinth was collected from Singanallur Lake (Coimbatore), retted, sun dried and chopped into two centimeters pieces and composted. The powdery coir waste was purchased from Universal Hydraulics, a coir briquetting industry (Coimbatore).

The initial C/N ratios were tested. At the end of 60 days of composting, the carbon and nitrogen levels showed that the selected wastes were complex in nature and slow in decomposing, in comparison with willow waste. Thus, similar cotton wastes were focused for the study. The white poom, blue poom and cotton dust samples from a denim industry in Ahmadabad, India was experimented. The raw sample from the industry was analyzed and decomposing trial study was done. Two among the three mentioned wastes showed very good results in composting.

Thus, to conclude, the poly manure from coir, jute and water hyacinth were different while comparing with the rate of composting with willow waste. Whereas, cotton processing wastes, namely cotton dust and white poom, were found to be composting faster than willow waste. The further study was built on these inferences.

3.2.2. Pilot study for production of non- woven products

Discontinuous fibers are spun into yarn to be used for weaving or knitting purpose, but too short fibers are engineered using non woven technology that does not require fibers as yarn, (http://www.inda.org/about-nonwovens/). Hence willow waste with cotton and trash can be made into non woven material.

Webbing is the preliminary step in any non-woven technique. Thus, willow waste was subjected to carding and webbing in the TIFAC Core (Technology Information Forecasting Assessment Council) of the Kumaraguru College of Technology, Coimbatore. The combs used in carding machine were jammed with the accumulated trash and apparently, the web was discontinuous and weak (Plate 2). Thus it was implicit to conclude that webbing for non-woven production using willow waste was not feasible.

Thermal bonding techniques of non woven making cannot be applied, due to the dry nature of the selected waste and it will eventually be burnt. Stitch bonding technique was not an economical solution in this case. Needle punching, another non woven technique, required a base fabric, that will hold the waste, on which, the willow waste will be interlocked. This technique was not practically viable.

3.2.3. Pilot study on conversion of willow waste into paper

Literature showcase that waste materials with lignin, can be used as raw material for paper making process (Kumar,*et al.,* 2013). The studies conducted by Ghosh and Pan., (2009) confirm the making of handmade paper from jute resides, based on this willow waste was also made into handmade paper. In order to check the effect of lignin from willow waste, no binders were used in this pilot study. Based on studies by Studley (2014), fifty kilograms of willow waste was taken, soaked and grounded into a fine paste using domestic grinder (Plate 2). With the addition of water, the diluted slurry was made to settle on a wire mesh and the resultant board was assessed. A performa (Appendix 2) was prepared and given to 50 students mastering in Textiles and Fashion Apparel in Avinashilingam Institute of Home Science and Higher Education for Women, Coimbatore (Tamilnadu, India) to access the characteristics of the prepared paper. This included general appearance, smoothness, texture, color and luster.

From the analysis it was agreeable that, 98% of the subjects felt the paper was weak and needed a binder. The rating also prove the prepared paper to be less appealing due to poor color, smell and texture as stated by 98, 95 and 95% of subjects respectively. Thus based on the pilot study results the use of industrial machine to grind the coarse willow waste and binders for internal and external reinforcement was used for the preparation of handmade papers in Phase III.

3.2.4. Pilot study to convert willow waste into wiping sheets

Wastes with lignin and cellulose are found to be good in wetting behavior and moisture absorption (Jimenez and Bismarck, 2007). The gushing issue of oil spills in gulf was suggested to be resolved by bio-sorbent properties of cotton and coir blends which form a lignocellulosic material. The phenolic groups in lignin are proved to aid in absorption of liquids (http://www.innocentive.com). As a solution to the same oil spill issue in gulf, a team of scientists with Ramkumar S. S., Manager, Non wovens and Advance Materials Laboratory (Texas Tech University, USA) confirm the property of raw cotton, to hold up to 30 pounds of crude oil and suggested sorbent sheets to pull oil from seas (http://www.acs.org and http://today.ttu.edu). Cellulosic materials along with the non-bio-degradable polypropylene are the common raw materials found in commercially available wipes (http://www.essentraporoustechnologies.com).

It is also quite evident that there is a need to manufacture unique low cost wiping materials. With this as an objective, melt blown synthetic thermoplastic microfibers were treated with a wetting agent, pattern bonded to incorporate strength and abrasion resistance, while absorbing water and oil without streaking (Meitner, 1985). This is the existing technique known to industries, but processing of organic wastes into wiping cloths, and incorporation of binders and adhesives will aid in creating an eco-friendly wipes. In this scenario, an experiment to convert cotton waste rich in cellulose (absorption) and lignin (strength) into a bio-degradable wiping cloth, will pave way for more researches and refinement of successful product in a commercial scale.

In view of the above researches, the willow waste was decided to be made into wiping sheets. Universal hydraulics (Coimbatore, India) was approached. The pressure (P) to be applied was calculated using the formulae.

P= f/a, where 'f' is the force and 'a' is the area. The hydraulic pressing machine exerts 20 tons of weight and in order to make a sample of dimension of area = 100 cm^2 and pressure of 200-300 kg/cm^2. Based on this, a die was prepared.

Willow waste was made into wipes along with coir and water hyacinth (Plate 3). Observations showed that the natural lignin present in willow waste, does not give 100% compaction. The samples were weak in nature. The un-uniform particle size of willow waste along with coir, water hyacinth was identified as a drawback. Hence the investigator felt the need for grinding and formulating the samples with the similar wastes like paper sludge. Based on these inputs, prototype of willow waste wiping sheets were made in Phase III and tested for its cleaning properties.

3.2.5. Pilot study for preparation composites

Lignocellulosic wastes with a thermoplastic polymer have a rising importance in various fields like automobiles, aircraft, home textiles, agro and geo textiles remarks Mtui (2009). Such products have many advantages like biodegradability, mechanical strength; thermal stability electrical conductivity and recyclability of such composites are very good. As mentioned by Castro, *et al*., (2012), use of additives help in increasing the mechanical strength of the composites. Fiber Reinforced Polymer (FRP) Composites, a subclass of composites are materials that are made of fiber and a matrix. This combination helps in compaction, smoothness and reducing overall cost (Tudu, 2009). Thus the investigator chose to make willow waste composites using natural and synthetic resin and compare its properties.

3.2.5.1. Selection of resins (natural and synthetic) for composite making

The study conducted by Veluraja,*et.al.,* (1998), in preparation of cellulosic rich, sisal-agave composite using tamarind gum, explains the strong bond between xyloglucon of tamarind and the hydrogen of cellulose. Hence the tamarind gum was selected for the study to make composites using the selected cellulosic trash, willow waste. One kilograms of TKP in powder form was purchased at Rs. 300/kg from Isckon traders, Krishnagiri (Salem, Tamilnadu) (Plate 4). Addition of polypropylene to lignocellulosic materials was found to improve their sound absorption, dielectric and thermal stability properties, compared to 100% polypropylene (Markiewicz, et al., 2012). Hence polypropylene was used as the synthetic resin. It was purchased from Zenith fibers, Surat, in the form of staple fibers and as sheets.

The TKP in powder form is mixed with willow waste and molded into a composite. Similarly staple polypropylene fibers were mixed with willow waste and made into composite boards. The powder form of TKP was found to be ineffective and they tend to burn off faster than reinforcing with the willow waste. Thus the TKP gum was then made into a paste and mixed with willow waste. The composites made of polypropylene fibers mixed with willow waste, were found to be patchy and not uniform. Hence, the polypropylene sheets were kept to sandwich the willow waste and the resultant composite boards were found to be satisfactory. The time, temperature and pressure used in the process were optimized for further study.

PHASE III

3.3. Development of innovative sustainable products based on pilot studies using willow waste

The selected willow waste was made into five sustainable products namely bio-manure, handmade paper, absorbent wipes, composites and briquettes based on inferences from the pilot study carried out in Phase II.

3.3.1. Bio-conversion of willow waste into bio-manure

The investigator chose on bioconversion of the selected willow waste using two decomposing sources and to further test the efficacy of the resultant bio manure in real field conditions.

3.3.1.1. Selection of decomposing sources

Studies on composting prove cow dung as an effective decomposing source and as a booster in organic waste management (Adegunloye and Adetuyi, 2007). This was further strengthened by the reports of Kaushik and Garg, (2004) on vermicomposting textile mill sludge using cow dung and earthworms. Based on their recommendations, willow waste was chosen to be decomposed using cow dung. Hitherto the merits associated with cow dung, its cost effectiveness and availability in urban areas pose a threat in large scale and in real time application. Hence a need to find another decomposing medium was felt noteworthy by the investigator. As an alternative remedy, Effective microorganisms (EM) technology was identified from the literature. Dr. Teruo Higa, a microbiologist and farmer, Japan, formulated this microbial consortium consisting of beneficial microorganisms and investigated its application in waste management and organic farming (Higa, 1989).

In view of the above facts both cow dung and effective microorganisms were selected to decompose willow waste.

3.3.1.2. Collection and preparation of the selected decomposing sources

Based on the Crop Protection Guide, TNAU, (2012) the selected decomposing sources were prepared for composting. Twenty kilograms of cow dung was purchased from a cattle farm in Singanallur, Coimbatore (India) for INR 10/kgs.

This was further diluted into cow dung slurry using two hundred liters of water and kept ready for immediate use (Plate 5).

Similarly, to prepare the effective microorganisms, the second decomposing source, two liters of stock solution was purchased from Sri Raam Biotech, Coimbatore (India) for INR. 250/ L. This was subjected to fermentation by mixing with two kilograms of powdered organic jaggery. To this liquid, twenty liters of chlorine free water was added (Plate 5). The container was closed with a lid and kept away from direct sunlight. The fermentation process began and carbon-di-oxide was expelled. Care was taken to release this accumulated gas, once in two days in the first week, and later once in a week. At the end of fourteen days, the solution turned into a dark brown liquid with fruity odor and bubbles on the surface. In normal room temperature, this solution was active up to three months. One liter of this solution was diluted with twenty liters of water and used for the study (Alamelu, 2006).

3.3.1.3. Collection of materials required for composting

Based on the nature of the selected waste, quantity to be composted and available area for composting, the ICRISAT model, suggested by Nagavallemma *et.a*l, (2004, which uses cement rings as composting bins, was adopted for this study. For this purpose, six cement rings of dimension 120 centimeters by 30 centimeters were purchased to make two separate composting bins to hold willow waste with the selected decomposing sources. Hay, barrels, jute sacks, jute roll, buckets and barrels, required for the study were also sourced and transported to the field for study (Plate 5).

3.3.1.4. Composting of willow waste

The purchased willow waste from OE mills (Singanallur) was transported to the composting field in Annur, Coimbatore (India). Unlike the pre-processing involved in composting of organic wastes, willow waste was directly subjected to the decomposing trails. For the field study, approximately 30 kilograms of bio-manure was required, considering this, hundred kilograms of willow waste was subjected to composting. Using three rings placed over each other, two separate composting bins were designed. Hay was the base layer. Over this, willow waste was layered for one foot height. The respective decomposing sources were added in the bin and thoroughly mixed with that quantity. Similarly, the next layer of willow waste and decomposing source were mixed and layered. This was repeated till the brim of both the rings. Finally jute sacks were used to cover the bins and commence the anaerobic composting of willow waste in both bins (Plate 6).

Generally composting requires the organic matter to be wetted completely (Ladan, 2014). During the experiments, it was observed that the willow waste was not wetted instantly and requires extra watering to spread the decomposing source.

This may be due to the dry nature of the waste and presence of wax content in cotton and trash particles. The composting bins were kept away from direct sunlight to trigger and help the microorganisms act on the organic waste and decompose it. It was found that the temperature of the composting bin initially increased due to the decomposition which makes the pit dry. The temperature and moisture of the composting bins were maintained by watering them periodically.

During the first four weeks watering was done once in three days and later once in a week. The heat released during composting was due to release of methane gas from composting and it ceases once composting was done. The contents of the bin were overturned at the end of 30 and 45 days for better composting results. The composting bins eliminated foul decomposing odor and also signs of fungal colonies were observed. Natural cellulose feeders like worms and termites were also seen, ensuring natures assistance in the decomposition process (Plate 7).

After sixty days, the compost turned brown with earthy smell. To test the compost parameters, sample from bottom and middle layer of the composting bin, were collected and tested for pH, carbon, nitrogen, C:N ratio, phosphorous and potassium. The reduction in C:N ratio, from the test results of compost (as in 4.2.1) confirmed the end of composting.

3.3.1.5. Vermicomposting of willow waste

Vermicompost is defined as black gold and organic manure produced as a nutritious vermicast by the action of earthworms. The art of vermicomposting is eco-friendly, consumes low energy with a recycled non-toxic biological product that enriches the soil quality by changing the physiochemical and biological properties. For the chemical polluted sterile soils, the vermicompost will be a great boon (Nayak and Jain, 2014). The experiments conducted by Abdalla, *et al.,* (2012)

showed that the addition of earthworms to the organic waste accelerates decomposition and helps in mineralization of organic matter. Accordingly, willow waste composted using both cow dung and effective microorganisms were subjected to vermicomposting.

3.3.1.5.1. Selection of earthworms for vermicomposting

Experiments on organic wastes, conducted by Kamineni and Sidagam, (2014), emphasize the benefits of using *Eisenia fetida* in transforming waste into bio-manure. As a non-burrowing earthworm, these act on the upper layer of the waste. In a day, it can consume 90% of organic waste and 10% of soil. The tolerance level is much better to other burrowing types, where it can sustain at temperatures ranging from 0°- 40°C and moisture between 40 - 45%. Based on these traits, *Eisenia fetida* was selected for vermicomposting willow waste.

3.3.1.5.2. Experimental design

Four kilograms of *Eisenia fetida* were purchased from H5 organics, Coimbatore (India) at a rate of INR. 250/kg. (Plate 8) Two kilograms were released in each of the two composting bins. Vermicomposting begins with the introduction of earthworms and watering was stopped. The optimum temperature for *Eisenia fetida* was 25°C, however it was reported to survive even in extreme conditions (Garg and Gupta, 2011). In the study, the temperature was maintained at 25 - 35 °C and moisture at 60%. Intense care was taken to protect the earthworms from escaping and being attacked by pests and reptiles from the nearby farm area. This was done by sealing the brim with jute sacks. At the end of 14 days, after the introduction of earthworms, granular vermicomposts were seen on the surface of both the bins. This was periodically collected and stored. As the level falls down, the cement rings were removed and kept aside. The collected vermicomposts from both the bins were stored in separate clean containers. Wetted sacks were used to trap the nutrients along with moisture. Finally, the collected vermicompost was sieved to remove earthworms or any other foreign materials like plastics, dry twigs, stones, dead insects. At the end of the study, fresh cow dung was kept in lumps, overnight, to collect the earthworms for further use. The grouped earthworms were washed in a bowl of water and separated to be added to fresh waste.

3.3.1.5.3. Testing of manure

The resultant manure was weighed to calculate the recovery percentage of willow waste decomposed using cow dung and effective microorganisms. The sample was taken from the middle and bottom layers to be tested for pH, moisture, carbon, nitrogen, phosphorous and potassium.

3.3.1.6. Field trials to assess the efficacy of the bio manure on cotton crop

Every crop requires nutrients in the form of fertilizers and pesticides to help them grow, fight against insects and give better yield. Unfortunately, with the invasion of such synthetics and overuse among ignorant farmers, serious health issues are aloud now. Thus organic farming has become the need of the hour. The compost when applied to plants helps in increase growth, resistance towards disease, improves the drainage and water retention property of the soil. It prevents soil leaching, increases the water holding capacity and organic matter content of the soil. The slow release nature of compost helps in long term benefit to the soil (Pace *et al.,* 1995). Based on the above mentioned facts, the efficacy of the bio-manure from willow waste using two decomposing sources was tested in real field conditions.

3.3.1.6.1. Selection of site and testing of soil

A farm land, twenty five kilometers from Coimbatore City, in Annur was selected for the study. It is important to test the soil in order to access the fertility, and calculate the fertilizer recommendations. The composition of soil varies from place to place and hence sample chosen, was a representative of the selected area (Soil Testing in India, 2011). The litters on surface of the soil were cleared, and a V shape slicing of soil from surface to ploughing depth was made. Using a spade, 1-2 cms of soil was sliced. The soil samples on the blade were collected in clean cloth bags and mixed thoroughly. This was further spread uniformly and then divided into four quarters. Two opposite quarters were rejected and rest of the soil was mixed and kept. The process was repeated till half kg of soil was collected. (Soil Testing in India, 2011).

3.3.1.6.2. Selection of cotton variety and purchase of seeds

Cotton, the major crop in India, contributes to 75% of raw material used for textile industry. It consumes about 16% of the fertilizers produced in the world and hence defined as the dirtiest crop (Organic Trade Association, http://www.ota.com). In India, cotton cultivation uses 45% of the fertilizers. (http://agritech.tnau.ac.in/bio-tech/biotech_btcotton_env. html). The major reason to use such strong chemicals especially on cotton is due to the pest attack on cellulose rich cotton. With the intervention of biotechnology and genetical modification of the cotton to be resistant against pests, the farmers have now shifted to Bt cotton. Now 34% of crop land and

45% of world's cotton production is Bt cotton, (www.ota.com). India is expected to be the world's largest Bt cotton grower as quoted in the September 04, 2014, article in *The Hindu,* Business line newspaper. It is reported that with the invasion and benefits of Bt cotton, the cotton sowing acreage has increased from 115 lakh hectares (2013-14) to 122.5 lakh hectares this year (2014-15). Hence to reduced use of insecticides, increase eco-friendly cultivation without compromising yield is important. Since 2002, with the import of Bt cotton, the acres cultivated is found to double every year. The overall yield is also quoted to have increased upto 30-40%. Reports confirm that there is 50-110% profit returns while using Bt cotton (Debyani and Neeta, 2012). Bt cotton prevents crop failure, giving better yield and in improving the economic condition of the farmer (Prasad, *etal*., 2009).

Considering these facts, The Kaveri Jackpot, KCH 15K 39 BG II, Bt cotton from Kaveri Seeds Company Ltd., was chosen for the study. It was also popular among the farmers in the selected area (Annur, India). This variety is also approved for commercial cultivation in India, in May 2007 by the Central Institute for Cotton Research (http://www.cicr.org.in).

3.3.1.6.3. Design layout of field studies

Randomized block design (RBD) is an experimental design in statistics in which different treatments are distributed in random order in the selected block or plot (http://www.merriam-webster.com). This is done to reduce the variability within the blocks compared to variability between the blocks. The samples within each block are then randomly assigned to the treatment. This is done to reduce variations within blocks compared to variations between the blocks (http://stattrek.com/statistics). Since the soil properties are not uniform, randomized block design was chosen to get approximate results on the effect of the prepared bio-manure. Based on this randomized block design consisting of six treatments in four replications were designed as shown in Fig. 1, the different colors represent different treatments and each horizontal row representing a block.

T4R4	T6R4	T5R4	T1R4	T3R4	T2R4
T3R3	T5R3	T1R3	T2R3	T4R3	T6R3
T1R2	T2R2	T3R2	T4R2	T6R2	T5R2
T6R1	T4R1	T2R1	T3R1	T5R1	T1R1

TREATMENT 1	Willow dust bio-manure using cow dung
TREATMENT 2	Willow dust bio-manure using EM
TREATMENT 3	Both chemical and green manure
TREATMENT 4	Green manure – MSW compost
TREATMENT 5	Chemical fertilisers as RDF
TREATMENT 6	Control

Fig. 1 .Randomised block design of the field for testing the effect of prepared bio-manure

The list of treatments include (1) willow waste bio-manure made using cow dung, (2) willow waste bio-manure made using effective microorganisms, (3) supplement including chemical and green manure, (4) commercially available green manure, (5) recommended dose of chemical fertilizers and (6) control treatment. (Plate 9)

3.3.1.6.4. Preparation of land for sowing of cotton seeds

The chemical fertilizer and manures required for the study were purchased from Mudaliyar and Sons (Coimbatore, India). Based on the crop requirement the fertilizers were added as basal, during sowing and as top dressing. The additive, rock phosphate was purchased and added to the prepared manures based on the deficiency.

The selected land was prepared by removal of the unwanted weeds and stones making it ready for ploughing, (Plate 9). Drip irrigation was the mode of watering for the cotton crop and necessary arrangements were made. In the ploughed area, the seeds were sown in the second week of September 2013 based on the randomized block design layout. The spacing was 75 cm between rows and 30 cm between plants, as stated in Crop protection guide - TNAU, (2012) and by

Deho, *et al*., (2012). Each hill was given two seeds and the end to of two weeks the plant with good stand was retained.

3.3.1.6.5. Cultivation of KCH 15K BG II using the different treatments

Randomly, one plant in each block was tagged and observations were recorded from that sample. The germination, flowering, number of leaves and branches from each treatment was observed and recorded. Growth observations were recorded at 60, 90 and 120 after the days of sowing (DAS).

As per the instructions mentioned on the seed packet, the given Non-Bt cotton seeds were also sown as border plants. Approximately, an area of 2000 (100 x 20) square feet or 0.018 ha was used for the experimental study. The gross dimension of one individual block (17 x 2 feet) was 34 square feet or 0.000315 ha. The recommended vermicompost was 500kgs/ ha (TNAU, 2012). Based on the nutrient recommendations of the cotton crop, one block was estimated and given approximately 158 grams of vermicompost and green manure treatment used in the study. Due to the slow release of green manure and composts, they were given as basal supplements, before sowing. However, chemical fertilizers were applied thrice: during sowing, then on the 45th and 75th days after sowing (DAS). With the second dose of chemical fertilizers, the weeds were removed. Thinning was done twice, on the first day and once in the second month of the crop.

3.3.1.6.6. Harvesting of kapas

Approximately after 42 days (six weeks) the cotton squares were seen. During the 45th to 50th day the flowers appeared. By the 90th - 110th day, buds were seen in the plants. After three weeks from the appearance of buds, thick creamy yellow flowers appeared. They turned into pink, red and the petals fell off in three days. The ovary was swollen and ripened into a big green pod with 3 to 5 cells, which later becomes the cotton fiber. This maturity happened between 55th to 80th days of sowing. On the 120th to 125th day, the kapas begin to appear and after 130 days, yield reached saturation. After 160 days there was no yield.

(Plate 10 and 11) Every week, the cotton fibers were plucked, weighed and stored in clean labeled paper bags.

3.3.1.6.7. Ginning of kapas for cotton fibers

Ginning is mechanical process that separates cotton lint from seed (Narasimha, Sridevi, and Reddy, 2011). The cotton kapas obtained from different treatments in the field were subjected to ginning in KG ginning unit, Annur, Coimbatore. The lint and seeds obtained from each treatment were collected in separate clean cloth bags and labeled. (Plate 12)

3.3.2. Preparation of handmade paper from willow waste

Based on the composition analysis of willow waste in phase I, and pilot study conducted in Phase II, investigator was interested in producing handmade papers from the selected willow waste. The lignin content in plant source, serves as a binding agent in paper making (ASTM, 1963) and hence paper was made.

3.3.2.1. Selection of paper making unit

Since the basic objective of research was sustainability, it was important to identify an eco-friendly handmade paper making unit. Jyothi Specialty Paper making unit, Erode, (Tamilnadu, India) is one industry in the south with complete effluent treatment plant using eco-friendly techniques which is also eco-certified by the Khadi Village Industries Corporation (KVIC). Considering the above mentioned facts the investigator selected Jyothi Specialty Paper making unit to prepare handmade paper from willow waste.

3.3.2.2. Production of handmade paper from willow waste

Sizing is done so that the paper is prevented from penetration of water and becomes hydrophobic. The study proves that rosin had no effect on the strength properties of the handmade paper and also application of wax emulsion on the surface hampering the quality of paper (Hossain et al., 2009). In view of the above researches washing and internal sizing was considered essential to increase strength and improve the hydrophobic properties of the prepared paper. Rosin and alum are commonly used to improve the porosity, resistance to wind and help in printing. Experimentation, on jute fibers using natural resins like starch, cationic starch, a low dextrin, gum arabic and guar gum was done. The results showed that strength and other properties improve while using natural resin, the appearance and strength is good (Ghosh and Pan, 2009). Hence rosin and alum were used for this purpose.

Rosin is a common water repellent compound (Silvestre and Gandini, 2008), which helps in enlarging the fiber to five folds higher than its actual size. In a non iron vessel, 1.5% rosin soap as solution was boiled with caustic soda (added as drops) in the ratio of 3:4. After thirty minutes, 3% alum was added to the mixture and stirred for five minutes (Asaduzzaman, *et al*., 2010). Alum acts as a water purifier, as an agent to maintain pH in addition to swelling and enlargement of the material and resulting in increase strength of paper state Chauhan and Sharma, (2014) and Hossain*, et al*., (2010). Hundred and fifty kilograms of willow waste was checked for the presence of any foreign materials and kept ready. In order to make handmade paper from willow waste a binder like baggasse or wood pulp is definite to reinforce the fiber matrix better (Personal Communication, Velayutham, Coimbatore, 05 November 2012). For reinforcement, fifty kilograms of unbleached cotton knit waste was purchased from Tirupur District (Tamilnadu, India) at INR.60/kg. This was cut into one centimeter pieces in a rack chamber machine and then passed into an industrial beater with 960 rotations per minute. The material binder ratio was 3:1. It was soaked for six hours and ground into fine slurry and then mixed with willow waste to produce handmade papers Shi,*et al*., (2010) and Ashori, (2006). The thickness of the paper was adjusted using the control knob. Willow waste handmade paper was made in it natural shade and no dyes were used. The collected sheets were sun dried. (Plate 13)

3.3.2.3. Calendaring of the prepared handmade paper

The handmade paper were kept as layers in between aluminum sheets and calendared. Two cylinders that can exert pressure of six tons were used. Six sheets were used at once. The calendared sheets were thin and had extra shine. Further, the edges were trimmed in a cutter and packed.

3.3.2.4. Value addition on the prepared handmade paper

Since the handmade papers were dull in color, the investigator decided to increase the value through dyeing and printing. Further it was decided to fabricate utility products using the enhanced handmade paper.

3.3.2.4.1. Dyeing and printing

An intensive exploration of natural dyes for handmade paper making Saakshy.*etal*., (2013) suggests the possibility to dye and print the prepared handmade paper. Hence the investigator underwent training in Aranya and Athulya, a group of TATA in Munnar (Kerala, India): (Appendix 3a and 3b). In the course, natural dyes, namely Indigo and madder were used and block printing, screen printing and resist dyeing techniques were followed to enhance the prepared handmade paper from willow waste.

The investigator sketched few designs for block printing and screen printing and later prepared wooden blocks and nylon screens. For resist printing the prepared handmade paper was placed on the wooden frames. The perforated design sheets where placed on the paper, care was taken to remove wrinkles. The printing paste (indigo) was poured over the design sheet and squeezed evenly from top to bottom using a rubber roller. The sheets were removed carefully. The printed handmade paper was removed and sun dried.

3.3.2.4.2. Preparation of value added utility products

In developed countries paper bags are the only option to carry away the purchased goods from any store. In India the awareness is less and now with the widespread understanding and ban on plastic bags, handmade paper bags foresee huge market potential (Atul Kumar, *et al*., 2013). So it is necessary to bring in paper bags that are both attractive and can carry weights.

The research paper by Prendergast, *et al*., (2001) highlighting the insights from consumers perception on shopping bags, this lead the study forward. Hence, the handmade paper made from willow waste was also converted into a carry bag. The prepared handmade paper was cut into required sizes, folded and the edges were sealed with tapioca starch. Banana or hemp fiber ropes can be used as a handle to paper bag (Mukherjee, n.d.). Since it was not feasible to buy small quantity of these, woolen ropes were used in the study. Further, the bags were screen printed

with unique and trendy designs created by the investigator. The printing was done using organic dyes purchased from Archana Academy Textile Printing Institute, Chennai, (Tamilnadu, India). (Plate 14 and 15)

Pineapple leaf fibers when used for making into fabrics result in pima fibers as waste, that are used in the production of handmade paper. The author showcase a series of products like paper pouch, picture frame, gift box, bonbon box, jewellery box, scrapbook that was made using the handmade paper from pima fiber. Nonetheless, these products are very attractive and have huge domestic and international markets (Espeso, 2014).

Considering the above facts, the easily portable, usable and recyclable properties of handmade paper are tapped for exploration of various products for daily life by the researcher. Many products including lamp shade, files, pouches, holders, envelopes, photo album, wallet, gift boxes, flower vase, jewel box, cake box were made using plain and resist dyed handmade paper. They were prepared by folding, cutting and finally sealing using tapioca starch and glue. Some of the products were also enhanced using dried flowers for decoration, (Plate 16-24).

3.3.2. Preparation of absorbent wipes

To prepare absorbent wipes, paper sludge which is having similar composition to that of willow waste was identified and purchased from GVG paper mills, Pollachi. In a semi dry condition this paper sludge had excellent compaction with willow waste. It was explicable that the paper sludge along with willow waste can be an effective cleansing material. The sample once made was hardened and there was no drape. Thus it cannot be made into a fabric-like cleaning wipe.

Since the handmade sheets were already made in a similar way, the sheets were cut into a 15 cm x 15 cm squares and used as absorbent wipes. (Plate 25)

3.3.3.1. Functional finishing of wipes

Finish is the application of any substance to the fabric to enhance its properties.

3.3.3.1.1. Selection and preparation of functional finishing agent

Since the prepared handmade paper in wet condition was able to act as absorbent wipes, the investigator planned to impart functional finish with natural agents for finishing the handmade paper which could be used as absorbent wipes.

Aloe vera and lemon peel show strong cleansing (Abhijit, 2012), (Pagan, *et al.,* 2011), and antimicrobial properties, [Parashar,et al., (2014) and Dhanavade and Jalkute, (2011)], and hence selected for the study. Mature aloe vera was collected and washed with clean water. The colorless parenchymatous tissue, aloe gel was scrapped out carefully. The collected plant gel was weighed and mixed with 100ml of ethanol, then left for 24 hours in a shaker incubator. The extract was strained and stored in the refrigerator at 40°C, (Stanley, *et al*., 2014). In a similar way lemon peel was collected, ground into a paste, mixed with ethanol and stored in cool condition, (Nisha et al., 2014).

The antimicrobial solution was prepared by mixing aloe vera and lemon peel extracts in the ratio 1:1 with 8% citric acid as binder (Chandrasekar, *et al.,* 2013). This was poured in the padding mangle and finished at pressure of 1kg/cm^2 (Pattanaik. and Ray. 2014). The handmade papers made from willow waste, imparted with cleansing and antibacterial property were tested for its physical properties and performance.

3.3.4. Preparation of composite using compression molding technique

Cellulosic wastes can be made into composites suitable for various applications like particle boards (Kowaluk, 2014), light weight laminate boards (Shibata, *et al.,* 2006), and sound proofing materials (Markiewicz et al., 2012), for varied industrial applications. Composites made from natural renewable resources, are called bio-composites. In an era of green consumerism, these hold a promising material for present and future market, due to its, varied advantages than the conventional petroleum based products. The ease in production, utilization of less energy, mechanical properties and low density are some of the merits in bio-composites (Mitra, 2014).

Thus a study to characterize composite made from willow waste was planned. Various natural and synthetic resins were experimented with fibers and the reports confirm the increase in tensile, bursting strength and folding endurance number. (Ghosh and Pan, 2009). Therefore a natural gum and synthetic resin were sourced to be used as binder and composites made were compared. The willow waste purchased was taken to the Technical textiles laboratory of Kumaraguru College of Technology, Coimbatore.

3.3.4.1. Sourcing of natural gum

Tamarind kernel powder (TKP) was made by processing the seed coat of *Tamarindus indica*, (Sumathi and Ray, 2002). This is insoluble in cold water (http:// www.niro.com/niro/cmsdoc.nsf/ webdoc/webb7lajmy). Reported to be a replacement for starch, TKP has varied application as an excellent binding medium in textiles. Compared to similar natural gums, this has better thermal stability and comparatively cheap (http://www.chemtotal.com/tkp.html). Hence two kilograms of tamarind kernel powder was selected as the natural gum.

3.3.4.2. Sourcing of synthetic resin

Polypropylene (PP) is the most commonly used synthetic resin in composite making. The application of PP in natural fiber composite production is suggested by (Mueller and Krobjilowski, 2003). The polypropylene staple fiber and sheets were purchased from Zenith fibers, Surat. The pilot study results of Phase II unveiled that polypropylene as core and as a surface layer had greater strength and compaction.

3.3.4.3. Making of composite

Willow waste was mixed with the natural gum in three different ratios, namely, 40/60, 50/50 and 60/40. Based on pilot studies conducted in Phase II, it was inferred that the ideal temperature and pressure to make willow waste composite using TKP as natural gum, was 100°C and 30kg/cm^2. Thus, the weighed and mixed samples were compressed in a compression molding machine. (Plate 27)

In the similar way, willow waste composites were made using synthetic resin, namely polypropylene in three ratios, 40/60, 50/50 and 60/40. From the results of the trail study conducted in Phase II, the temperature and pressure were maintained at 165°C and 40-45 kg/cm^2. Another insight from the pilot study was the significance of

using polypropylene as fibers in the core mixed with willow waste and PP sheets as top and bottom layer. The samples were made in this technique. (Plate 28)

Willow waste composites using TKP and PP were trimmed before testing with the aid of industrial cutting machine from Mechanical Engineering Department and tested in Universal testing machine in Textile Technology Department of Kumaraguru College of Technology, Coimbatore.

3.3.5. Developing prototype of boiler feed material

Textile is an energy intensive industry and bringing down the cost incurred for energy is essential. Wet processing involves dyeing, printing, washing, pre-finishing, drying and final finishing of textiles (http://www.forbesmarshall.com), that consumes huge amount of hot water (70 - 90°C) and steam (Abbi and Jain, 2006). Wooden logs are conventionally used to heat the huge boilers for this purpose. It was interesting to know that the ligno-cellulosic waste can be briquetted and its fuel properties were compared (Granada,et al., 2002). Thus the investigator chose to compress willow waste into briquettes.

3.3.5.1.Briquetting of Willow waste

Slow and steady burning is an important parameter for boiler feeding material. In the pilot study, willow waste was found to burn off rapidly. Hence 100% willow waste and 50/50 (v/v) in combination with coir was chosen to be experimented. The willow waste briquette was made in hydraulic pressing machine in Universal Hydraulics, Ganapathy, (Coimbatore, India). Binders were not used.

In ambient condition, the samples were briquetted in a manually operated hydraulic pressing machine with a capacity of five tonnes. Hundred kilograms of willow waste was compacted with the pump speed of 30mm/min. The compacted pressure ranged from 3.0 – 9.0MPa. A known pressure was applied at a time to the material in the die and was allowed to stay for 45 seconds (dwell time) before being released and the briquette formed was then extruded and labeled. Stop watch was used for purpose of timing. In a similar manner willow waste with coir was made into briquette. (Plate 29)

3.3.6. Nomenclature

The nomenclature of the samples taken for the study is as follows.

SAMPLE DETAILS	NOMENCLATURE
Willow waste	WW
Cow dung	CD
Effective microorganisms	EM
Willow waste compost made using cow dung as decomposing source	WWCCD
Willow waste compost made using effective microorganisms as decomposing source	WWCEM
Willow waste bio-manure made by vermicomposting the composted willow waste using cow dung as decomposing source	WWVCCD
Willow waste bio-manure made by vermicomposting the composted willow wastes using effective microorganism as decomposing source	WWVCEM
Integrated Nutrient Management, using both chemical manures and green manure	INM
Green manure (commercially available municipal solid waste compost)	GM
Recommended dose of chemical fertiliser for the cotton crop	CHE
Pre-sowing soil sample collected before sowing	PS
Willow waste wipes made finished using aloe vera and lemon	WWW
Polypropylene	PP

Tamarind Kernel powder	TKP
Willow waste composites made using synthetic resin polypropylene	WWCPP
Willow waste composites made using natural resin Tamarind Kernel Powder	WWCTKP
100% willow waste briquette	WWB
50/50 willow waste with coir	WWCB

PHASE IV

3.4. Evaluation of the recycled products made from cotton waste

The products developed by recycling and upcycling willow waste was tested for its performance properties and compared with similar products.

3.4.1. Testing the bio-manures obtained from willow waste

The bio-manure prepared using two decomposing sources namely cow dung and effective microorganisms were tested for manurial properties, like pH, electrical conductivity, carbon, nitrogen, phosphorous, potassium and C: N ratio in compost stage and after vermicomposting. These results were compared with bio-manures obtained from other wastes and with the standards set in organic cultivation.

3.4.2. Testing compost and vermicompost

The compost and vermicompost (WWVCCD, WWVCEM) were analyzed at the Department Environmental Sciences, Tamilnadu Agriculture University, Coimbatore, (India).

The pH was measured in suspension of 1:5 (soil: $CaCl_2$) according to Hendersont *et al.*, (2008), while the electrical conductivity (EC) 1:5 was determined in suspension (soil: water). The available phosphorus (P) was measured calorimetrically by the molybdenum blue method (Jackson, 1958). Nitrogen (N) was estimated by micro-Kjeldahl method (Association of Official Analytical Chemists, 1990) and OC was determined by Walkley and Black (1934) method. Dry washing was conducted to prepare the samples for the determination of calcium (Ca_2), magnesium (Mg_2), sodium (Na) and potassium (K) as per Chapman and Pratt (1961) instructions. Sample extraction was performed following the procedure of Perkin

(1994) for the determination of manganese (Mn), iron (Fe), copper (Cu) and zinc (Zn). Mineral nitrogen (NO_3-N and NH_4 þ-N) in compost and vermicompost were measured according to Bremner and Keeney (1965) procedure.

3.4.3. Comparing the yield of cotton kapas obtained and Ginning out turn (GOT) %

Ginning out turn (GOT) %, is the ratio of lint weight to seed cotton yield (cotton + seed) weight expressed in percentage (Shukla and Khakare, 2013). GOT was calculated for each treatment. The quality of cotton fiber from each treatment was tested using (ASTM D 5867, 2005), a High volume instrument (HVI) in Central Institute of Research on Cotton Technology (CIRCOT, Coimbatore). The mean weight of the kapas from each treatment was compared and inferences were drawn. As per the calculation given by Saleem, *et al*., (2011), the ginning out turn (%) was calculated as the percentage of ratio of weight of lint in sample to weight of seed cotton in that sample;

$$\text{GOT (\%)} = \frac{\text{Weight of lint in sample}}{\text{Weight of seed cotton in that sample}} \times 100$$

3.4.4. Comparing the soil properties before (pre-sowing) and after the study

Soil samples collected as stated in 3.4.1.6.1, before and after the study was tested for pH, electrical conductivity, cationic exchange capacity, carbon, phosphorous, calcium, zinc, iron, sodium, nitrogen, copper, manganese and magnesium as per the manual for soils in India [(UPASI, Ministry of Agriculture, (2011)]

3.4.5. Evaluation of cotton fiber properties using High Volume Instrument (HVI)

Span length is the extent exceeded by the stated proportion of cotton fibers and 2.5% span length corresponds to the length exceeded by, only 2.5% of fibers by number. (http://www.probrite.net/define.asp?def=Fiber%20length). Uniformity ratio UR (%) or Uniformity Index is the length uniformity of the fibers. It is considered to be very low if found below 76, low if the value is between 77 – 79, average if the value is between 80 – 82, high if it 83 – 85 and very high above 86. Micronaire (µg/inch) is a parameter that describes the fineness of cotton fiber. It is considered to be very fine if the values are below 3, fine if it ranges between 3.1 – 3.9, average if it is between 4.0 -4.9, coarse for values between 5.0 -5.9 and very coarse for values above 6. The tenacity is ratio of force by linear density that represents the fineness of the cotton fiber. The breaking point of a splice of cotton fiber is measured as the elongation (%). Yellowness of the fibers is denoted as +b values which are graded based on the Nickerson/Hunter scale. The reflectance of the cotton fibers are marked by the Rd values and higher values symbolize higher color grade. (http://www.uster.com/fileadmin/customer/Knowledge/Textile_Know_How/Yarn_testing/U_LabSystems_Description_of_al_quality.pdf) The whiteness index (WI) was calculated using Hunters formulae. [WI= Rd – 3(+b]) and true matt (%) represents the cellulose content in the fiber.

The cotton fiber qualities namely, 2.5 % span length, uniformity ratio, micronairre, tenacity, elongation, +b between treatments, Rd value, whiteness index and true matt was calculated using HVI (ASTM D 5867). These tests were carried at SITRA, Coimbatore and the results obtained from the study were compared with the standards.

3.4.6. Testing of handmade paper

The handmade paper made from willow waste was tested for weight, thickness, elongation, tearing index, burst, foldability, endurance number, water absorption and moisture content. The physical properties of paper like thickness, GSM, tearing strength, Cobb's value, water absorbency, folding endurance were measured in the standard temperature and humidity (Temp= 23± 10^0C, RH=50± 2%) for paper as per TAPPI standard test methods after conditioning for three days according to T402gm-93 procedure.

3.4.6.1. Test for weight (GSM) and thickness

Ten samples of 10cm × 10cm were taken from ten spots and measured using vernier caliper according to TAPPI T441om-97 method. The results were recorded in mm. Mass per unit area gives the weight of the sample. Using a GSM cutter, the sample was cut and weighed. Ten samples of 10cm × 10cm were taken and weighed in an electronic balance and the values were multiplied by 100 to get the weight of the sample in grams per square meter (Hossain, et al., 2010).

3.4.6.2. Determination of Cobb Value (water absorbency test)

As per the methods discussed by Hossain, *et al.,* (2010), three samples of dimension 12.5cm×12.5cm for each experiment were cut. Water absorbency was tested according to TAPPI T 441om-98 method.

Each sample was weighed first to the nearest 0.01g. A dry rubber mat was placed on the metal plates and weighed sample was placed on it. The dry metal ring was placed upon the sample and fastened firmly in place with crossbar to prevent any leakage between the ring and the sample. 100ml of water (23± 10^0C) was poured into the ring as rapidly as possible thus giving a head of 1.0± 0.1cm. The water was poured quickly from the ring after 110 seconds. The crossbar was taken out of the way by loosening the wing nuts promptly while holding the ring in position by pressing it down with one hand. The ring was removed carefully but quickly and the sample was placed with its wetted side up on a sheet of blotting paper resting on a flat rigid surface. Exactly at the end of the pre-determined test period (2 minutes), a second sheet of blotting paper was placed on top of the sample and the surface water was removed by moving a hand roller once back and once forward over the pad without exerting any additional pressure on the roller. The sample was folder with the wetted area in side and was re-weighted immediately. The weight of water absorbed in g/sq meter was obtained from the following formula:

Weight of water absorbed (Gm/m^2) = {Final wt. (g) - Conditioned wt (g)} × 100. Ten readings were taken and recorded systematically for further statistical analysis.

Test for tensile index and elongation

Tensile index is the maximum force required to rupture a test strip of standard width and is reported as N/m. http://www.iso.org/iso. The samples were tested for tensile index and elongation as per ASTM D828-97. Tensile Testing Machine, of the constant-rate-of elongation type was used to test the samples. Test specimens of dimension 2.54 ±0.5cm width and 25.4±0.5cm length without any creases, holes, wrinkles were clamped between the jaws of the instrument. Ten test specimens in each principle direction were tested and tensile strength and elongation was recorded.

3.4.6.3. Testing the tearing index

Elemendorf tear tester was used for evaluating tear resistance following ISO 1974. The force needed to disseminate tear through a paper is known as tear index. Four test pieces superimposed with specified pre-cut slit was torn by moving the pendulum at a fixed distance. The tearing resistance of the paper was determined from the average tearing force and the number of sheets comprising the test piece. The average tearing force was indicated in a digital display.

3.4.6.4. Testing the burst index

This was tested in Mullen tester. Ten samples cut from different parts of the paper were clamped between two concentric plates with a circular opening in the centre. The open space is the testing area. The area of the test pieces was kept wide enough to be securely clamped (ASTM, 1963). Pressure was applied from the underside by a rubber diaphragm which expands due to hydraulic pressure. It is defined as the one burst made on each side of each four specimens. In this manner ten readings were noted.

3.4.6.5. Determination of the folding endurance number

Folding endurance is very important in term of indication for durability and performance (Othman, et al, 2013). Folding endurance test is a measure of strength that a specimen holds under a constant tensile load. The folding endurance tester is a machine that tests the number of fold which a specimen can withstand before failure, under controlled condition. Ten specimens were subjected repeatedly to double fold through a wide angle kept under tension and the number of folds each sample could withstand was recorded.

3.4.6.6. Test for water absorption

The rate of absorption was tested using Byreck method. This is known as Klemm method for testing papers. The absorption was more when the rising was higher (Takahashi, et al., 2014). The test samples of dimension 1.5 cm were submerged perpendicularly in distilled water (20±2℃) and the rising height of water after ten minutes was noted on the graduated ruler mark. The rising height of the distilled water in the paper starting from 2.5 minutes to thirty minutes was noted.

3.4.6.7. Test for moisture content

Moisture is an important property to asset the printability, shrinkage, dimensional stability, physical strength and run ability of paper. Therefore following the ISO 287: 2009 the moisture content of the prepared handmade paper was calculated. Ten test samples were cut as per the specifications and weighed before and after oven drying at 105^0C.The moisture content percent was calculated and recorded using the formula

Moisture Content (%) =Initial dry weight - oven dry weight divided by oven dry weight into hundred.

3.4.7. Testing the effectiveness of the prepared wipes

The sheets made from willow waste were subjected to finishing with aloe vera and lemon in 50/50. The wiping sheets were further subjected to testing of pH, flushability, moisture management and absorbency tests.

3.4.7.1. Test for pH using aqueous

The pH of the willow waste wipes was tested by soaking the sample in distilled water for two hours and testing according to ASTM D2165-1994, (2012). (Campbell, 2003)

3.4.7.2. Test for pH using water

It is important to test the pH of willow waste wipes to know the acidic or alkali contents on the wipe sample. For this, 250 ml of distilled water was boiled and 10gms of the test specimen was immersed into it and boiled for another ten minutes. The sample was squeezed for excess water and kept aside. The pH was calculated using a calibrated pH meter (https://www.aatcc.org/Technical/Test_Methods/scopes/tm195.cfm). Ten readings were taken and recorded.

3.4.7.3. Sinking time (seconds)

Four samples of size one centimetre by one centimetre was taken one by one and kept on the surface of water taken in a 500 ml glass beaker. Sinking time is the time taken by the piece to sink beneath the water surface. This was measured and reported, (Ul-Haq and Nasir, 2012).

3.4.7.4. Water holding capacity

Evaporation rate or the water holding capacity of willow waste wipes was calculated by weighing the samples after being soaked in water. The resultant values help in understanding aeration, water penetration and water retention properties.

3.4.7.5. Moisture management test

The overall (liquid) moisture management capability (OMMC) is an index of the overall capability of a fabric to transport liquid moisture as calculated by combining three measured attributes of performance: the liquid moisture absorption rate on the bottom surface (ARB), the one- way liquid transport capability (R), and the maximum liquid moisture spreading speed on the bottom surface (SSB). The moisture management test was conducted using AATCC test method 195-2011 in the instrument SDLATLAS. Test specimens with dimension 8cm X 8cm was cut and kept inside the machine and analysed for top surface and bottom surface wetting time (sec), absorption rate, (%/sec), wetted radius (mm), spreading speed (mm/sec) and one way transport capability. Five readings were taken and based on the grades given, the samples were evaluated. The test for run-off time and porosity could not be tested on this specimen. According to the AATCC - 195 standards, it is graded as 0 -0.2 as very poor, 0.2-0.4 as Poor, 0.4 – 0.6 as good, 0.6-0.8 as very good and above 0.8 as excellent. The work wear clothes have minimum Overall moisture management capacity (OMMC) is 0.8. (http://www.nfpa.org/Assets/files/AboutTheCodes/1975/1975_F2013_FAE-SCE_FirstDraft_ballot.pdf).

3.4.7.6. Flushability

Flushability is also called as column settling test. The test for flushability using willow waste wipes was done in FLUSH 100, Flushability tester, Lenzing Instruments. Four samples were taken, and put in each of the plastic tube containing a known amount of test liquid. After a specific number of rotations, the size of the residual fragments was analyzed by means of a sieve box. The values including overall percentage of weight loss and the weight loss in sieve was measured for further interpretation. (http://www.lenzing-instruments.com/produkt.infos/flush100-2.pdf) (Plate 26)

3.4.7. Assessing the composites prepared using willow waste

The composites prepared using natural gum (TKP) and synthetic resin (PP) in three ratios namely 40/60, 50/50 and 60/40 were tested in Universal Testing machine for its tensile, elongation, modulus at 5% and flexural rigidity. The samples were cut in dimensions required for testing as per ASTM D 638-01. Ten samples of TKP and PP were prepared for testing the mechanical properties, namely tensile, elongation, flexural and modulus using Instron Universal Testing Machine (Hashmi, et al., 2011).The results were tabulated and compared.

3.4.8. Testing the fuel properties of willow waste as boiler feed material

The briquettes made using 100% willow waste and 50/50 (v/v) willow waste and coir were tested for the following properties in Bio-energy workshop, Tamil Nadu Agricultural University, Coimbatore and Heat lab, P.S.G. College of Technology, Coimbatore.

3.4.9.1. Measuring the dimensions and weight of each briquette

Each briquette was measured using an inch tape for its length, breadth and width. The sample was weighed on an electronic weighing machine. The average of ten samples was noted.

3.4.9.2. Determination of moisture (%) and ash content.

The moisture content of a compacted briquette is an important parameter that signifies the rate of combustion and heating value of the combustion gas. The briquettes were tested for moisture and ash as per the studies reported by Raju, et al. , (2014). To determine the ash content a previously weighed silica crucible (W_E), about one gram of sample was taken and weighed accurately (W_S). The crucible along with the sample was heated in a muffle furnace at 725 ± 25°C for nearly half an hour. The crucible was then cooled on a ceramic tile for few minutes followed by cooling in a desiccator to attain room temperature and then weighed (W_A). From the weight difference, the percentage of ash was calculated and noted for ten samples;

$$\textbf{\% of ash content} = \frac{\text{Weight of ash left}}{\text{Weight of sample taken}} \times 100$$

In order to determine the moisture percentage a previously weighed silica crucible (W_1), about one gram of coal sample was taken and weighed accurately (W_2). The crucible along with the coal sample was placed in a hot air oven at 110°C for one hour to remove moisture. It was then taken out and cooled in a desiccator to attain room temperature and it was weighed again (W_3). From the weight difference, the percentage of moisture was calculated and noted for ten samples.

$$\textbf{\% of moisture} = \frac{\text{Weight loss due to moisture removal}}{\text{Weight of coal taken}} \times 100$$

3.4.9.3. Estimation of calorific value

Calorific value is the quality index for fuel (Suhartini, Hidayat, and Wijaya, 2011). It is a qualitative measure for briquette, which is defined as the amount of heat generated during combustion of the material. The chemical composition and moisture content of the selected biomass affects the heat value or calorific value. The calorific value was estimated using bomb calorimeter (Sengar, et al., 2012). Ash is the unburnt material at the end of combusting fuel and its value is inversely proportional to the fuels calorific value. It affects the diffusion of oxygen and heat transfer to the surface of fuel and is expected to be low in a fuel material.

About one gram of the sample was weighed and placed inside the designated cup. The ignition wire was inserted between the electrodes. The wire was positioned just above the sample holder in such a way that it touches the sample. Two ml of distilled water was placed inside the bomb to absorb any acidic gases. The bomb was then closed and cap was hand tightened and purged with pure oxygen twice. Finally the bomb was filled with oxygen allowing the pressure to build up slowly to 25 atm. The ignition head was connected to the top of the bomb after which, the bomb was immersed into 2000 ml of distilled water placed in the calorimeter. The outside water jacket was filled with approximately 20 liters of tap water. After the setup was done, the samples (WWB, WWCB) inside the bomb were burnt electrically. The temperature rise of the calorimeter was directly read as ΔT from the display panel and was used in the calculation. After the experiment, the pressure inside the bomb was vented and the lid was opened and the vessel was cleaned. The gross calorific value of the fuel (GCV) was given by the equation.

GCV = (W +w') ΔT, Where, W is the mass of water in the calorimeter, w' = mass of bomb X C_v (bomb) and ΔT = net rise in the temperature of the water in the calorimeter.

However, the fixed carbon and volatile matter could not be tested due to the heterogeneous nature of the sample that gave varied results every time.

3.4.9.4. Determination of burning rate (g/min) and specific fuel consumption (g/liter)

Burning rate is defined as the ratio of mass of fuel consumed (g) to the total time taken (min) and specific fuel consumption is mass of fuel required to produce one liter of boiling water. It was calculated as the ratio of mass of fuel consumed to total mass of boiling water [Davies and Abolude, (2013) and Bharti and Awasthi, (2012)].

3.4.9.5. Thermogravimetric analysis (TGA)

The thermal decomposition behavior of the cotton and cotton/coir briquette was examined using Thermogravimetric analyzer (Q50 v20.13). Willow waste (46.04 mg) and willow waste/ coir (39.9 mg) was placed in two separate aluminum sample holders and heated from 28 °C to 1000 °C at 10 °C per min heating rate using nitrogen (flow rate of 25 ml/min) as sweeping gas. The theromograms obtained were subsequently analyzed to examine the thermal decomposition of the samples on a comparative basis (Nyakuma, *et al*., 2014).

3.4.9.6. Bulk density

The water displacement method was used to calculate the volume of an individual briquette. Ten samples were weighed and then coated with wax in order to prevent water absorption. In a suspension position the samples were submerged and weight of the displaced water was recorded as volume of wax coated briquettes. The volume of each briquette was calculated by subtracting volume of coating wax from the volume of waxed briquettes. The volume of coating wax was obtained by dividing its weight of the wax obtained by subtracting original weight of briquette from the weight of wax briquette by its volume (Sengar *et al*., 2012). The readings were noted for further calculations.

3.5 Statistical Analysis

The data collected from various evaluation parameters were statistically quantified to significantly conclude the influence of parameters on the experiments. Statistics aids in condensing the collected data, its analysis, comparison and interpretation, thus helping the researcher to come to a true statement about the influence of various parameters considered during the study. The data collected were presented in table, represented with suitable figures, graphs and statistically analyzed with suitable tools mentioned below.

3.5.1 Arithmetic Mean

Data tends to cluster near central value and Arithmetic Mean, which is the most prevalent used measure of statistics to represent the entire data in one value, so it was used for the study.

3.5.2 Standard Deviation

The observations marked as data will not be alike and there will be variations among them even though it was represented in a single value by arithmetic mean, unless all the observation noted were same. So it is essential to define the variability of the data. Measures of variation assist in understanding the vital characteristics of distribution. Standard deviation is the utmost vital and extensively used measure for studying variation. It fulfills the qualities required for a good measure of variation and is also free from faults.

3.5.3 CV %

The experiment study dealt with more than two groups of treatments, so to compare the variability of more than two series of treatments coefficient of variation was used. The series for which the coefficient of variation is greater is said to be more variable or controversially less consistent, less uniform or less stable (Gupta and Gupta, 2011).

3.5.4 ANOVA

The analysis of variance frequently referred to by the contraction ANOVA is specially designed to test whether the means of more than two quantitative populations are equal (Gupta, 2012). Since various treatments were used in the experiment, ANOVA was used to decide whether the mean qualities of the outputs of various parameters differed significantly.

3.5.5 F-Test

F test was used to find out whether the two independent estimates of population variance differ significantly or to know if they were drawn from the normal population having same variance.

3.6. Cost Analysis

In order to check the productivity and marketability of the prepared sustainable products namely willow waste bio manure, handmade paper, wipes, composites and briquettes a cost analysis was done in comparison with similar commercial products. This is presented in the chapter Results and Discussion.

Bio-manure: The fixed cost like cement rings, sacks, buckets, cow dung, organic jaggery, urea were purchased and their rates were noted. Transport cost was also calculated and recorded. This rate was compared with compost made form municipal solid waste and commercially available vermicompost, which included the cost of earthworms.

Handmade paper: The cost occurred during the purchase of raw materials like willow waste, knit waste, rosein soap, caustic soda and alum for the production of handmade paper from willow waste were noted. The transportation and production charges were added to the total cost from which unit price was calculated. This was further compared with commercially available handmade paper from other sources.

Wipes: The handmade papers made from willow waste were noted. Further the cost for finishing included purchase of citric acid (binder), aloe vera and ethanol. The rate per piece was calculated with the commercially available wipes and compared.

Composites: The cost on the purchase of willow waste, natural gum tamarind kernel powder and synthetic resin polypropylene were noted. Based on the purchased materials the cost was calculated and compared with commercial composite boards.

Briquettes: The cost of willow waste and coir was calculated and compared with commercially available briquettes made from other sources like agro waste and coal.