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On

"Compostable biopolymer coatings for improving functionality of molded pulp food packaging"

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in

PACKAGING TECHNOLOGY

By

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CANDIDATE'S DECLARATION

I hereby declare that the work carried out in this dissertation titled "Compostable biopolymer coatings for improving functionality of molded pulp food packaging" is presented on behalf of partial fulfilment of the requirement for the award of the degree of Master of Technology with specialization in Packaging Technology, submitted to the department of Paper Technology, Indian Institute of Technology Roorkee, India, under the supervision and guidance of Dr. Anurag Kulshreshtha, PPD, IIT Roorkee, India and Dr. Ruchir Priyadarshi, YASH Paper Ltd.

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CERTIFICATION

This is to certify that the above statement made by the candidate is correct to the best of my knowledge and belief.

Dr. Anurag Kulshreshtha Dept. of PPD, IIT Roorkee I extend my sincere thanks, reverence and obligation to Dr. Anurag Kulshreshtha for his through guidance at every step knows no bound, as for without their genuine help this dissertation could never take the present shape. I take this opportunity to express my deep sense of gratitude to him for his invaluable guidance, constant encouragement and abiding inspiration.

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I would like to express my sincere thanks to the Head of Department and faculty members of Department of Paper Technology for their valuable guidance and suggestions as and when required by me.

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Date: 24 May 2019 Place:

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Jagdish Chandra Arya

ABSTRACT

Paper is most widely used in packaging application. But it has some properties create hindrance to its use in the packaging application. Its characteristics of printability, recyclability and biodegradability make more use in packaging industries. It is also useful as per environmental concern, it replace the plastic packaging materials with the degradable material. The work has main purpose is to improve the properties of the paper using some biopolymer which are either home or industrial compostable. Here, we are increasing the properties of water vapour barrier, grease barrier and heat seal strength of the paper material using coating of biopolymer on it. Here, we are testing three types of method to improve the above mentioned properties.



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1. INTRODUCTION

Here we are using biopolymers as coating materials for the moulded pulp packaging. To reduce the use of the synthetic polymer using to increase the barrier properties of the packaging materials replacing them using the bio-polymer materials to increase the sustainability of the procedure and the bio-compostability of the material either home compostable or the industrial compostable. Nowadays, it seems that companies are more conscious towards the environmental concerns.so they are using biopolymer coating to get the barrier properties.

The main mission of food packaging is to maintain the quality and safety of food products during storage and transportation, and to broaden their shelf-life by avoiding unlikeable circumstances, such as hazardous microorganisms and their corresponding toxins, external physical forces, chemicals compounds, sunlight, permeable volatile compounds, oxygen and moisture. Currently, a large number of petroleum based polymers are used in food, beverage and foodstuff packaging industries, with consequent accumulation of non-degradable and non-recyclable waste materials and concerns over environmental issues. Thus, there is an increasing demand for bio-based raw materials in order to unravel the waste disposal problems to an assured magnitude.[1]

To achieve the particular property of the substrate materials we can use different methods and different way to achieve that property. Either the single biopolymer can achieve the property or we require two or three polymer mixing to achieve the desired properties. Also there is bilayer or tri-layer coating is required to get the required properties. Only one or two biopolymer cannot achieve all the properties so there is mixing of biopolymer to obtain that properties. Suppose if we require water vapour barrier with oxygen barrier, here we will search for the biopolymer materials which can be used to uphold the mentioned characteristics, which is either one biopolymer, two biopolymer or mixing of these biopolymers.

The biodegradable biopolymers can be classified in three main categories on the basis of their origin as follows:

- Biopolymers originated from agricultural resources, including polysaccharides (e.g. starch), ligno-cellulosic products (e.g. cellulose and its derivatives), proteins (e.g. whey and collagen), lipids (e.g. bee wax) and free fatty acids
- 2. Biopolymers achieved by means of microbial fermentation, such as pullulan and polyhydroxyalkanoates;
- 3. Chemically synthesized biopolymers using monomers attained by natural raw materials such as poly(lactic acid).[1]

1.1. Issue with the existing materials

The objective of my work is to provide grease and water resistance as well as water vapor functionalities to molded pulp packaging by spraying the biopolymer solutions after or before drying of the articles. Applied polymers shall not hinder the home compostability of the packaging. The existing synthetic materials using to get the water vapour barrier, gas barrier and grease are given below with their existing problem :

- 1. **EVOH** is hygroscopic and absorbs water at elevated relative humidity, and then loses much of its oxygen barrier performance Water molecules absorbed by EVOH at high RH are believed to combine with hydroxyl groups in the polymer matrix and weaken the existing hydrogen bonds between polymer molecules. As a result, segment movement becomes easier, facilitating the diffusion of oxygen and perhaps other gases.[7]
- 2. Although **PVDC** has good water vapour and oxygen barrier properties but its combination with the other materials cannot be recycled easily.[8]
- 3. Aluminium foil gas good water vpour and gas barrier property but it is not degraded to the environment. Here we can just do down-gauging. The materials we are using now are not biodegradable also not compostable to the environment which increases the landfill waste. The packaging industry aims to reduce to zero landfills.[2]
- 4. One of the sustainable alternatives that could be considered to deal with plastic waste is to develop bio-based and biodegradable plastic which utilize starch, cellulose, and poly lactic acid as raw materials for short-term use products.[2]

5. Recycling options are generally classified into primary and secondary recycling, while tertiary recycling is preferred for multi-layered plastics (MLPs) where separating individual layers is difficult and expensive. Most companies prefer MLPs as they are three times more waterproof, light-weight, reduce shipping volume, and help in increasing the shelf life of products.[2]

The main concerns with the existing materials using for the improvement of the barrier properties are recyclability and impact of materials on the environment. So, we can use compostable or bio-degradable materials for the short span to improve the properties of the packaging. Either the material is home compostable or industrial compostable is better for the environment and sustainable.

Plastic	Application
Polyethylene Terphthalate (PET)	Water and soft drink bottles, food jar
Polyvinyl Chloride (PVC)	Cables, plumbing pipes
High-density polyethylene (HDPE)	Shampoo bottles, packaging
Low-density polyethylene (LDPE)	Grocery bags, packaging
Polypropylene (PP)	Bottle cups, medicine bottles, chips pack
Polystyrene (PS)	Disposal cups, cutlery, packaging foam
Polycarbonate (PC)	Food packaging, electronic goods and defence gadgets
Nylon	Fishing nets, clothing, ropes
Aluminium Foil	Food and pharmaceuticals packaging, electronic item

Table 1: Major uses of plastic materials in industry.[2]

Film Type	Thickness (µ)	Oxygen Transmission cc/m ² /24 hours (100% oxygen) 25°C 45% RH	Water Vapour cc/m ² /24 hours 90% RH
Metallised Mylar	12	0.5	<1
PVdC Coated Polyester	12	8	14
Propafilm C28	28	10	5
Propafilm CR	28	25	4
Propafilm MG	20	2200	7
Propafoil (Metallised)	25	100	1.5
EVAL F	20	0.2*	75
EVAL E	20	1.8*	29
Extruded PVdC	20	3	5
Aluminium Foil	9	0+dependent on pinholes	0+

 Table 2: Materials used for increasing oxygen barrier and water vapour barrier.[9]



2. LITERATURE SURVEY

Here, instead of using non-biodegradable conventional polymer we will use the compostable polymer to increase the performance of the barrier properties on packaging. My objective is increase the water vapour barrier properties and grease barrier properties of the sample. So, here I am using replace the existing materials with the following biopolymers, either it can be done through the single layer coating of biopolymer or by the double layer coating of the biopolymer having different barrier properties :

- **2.1. Mixture of carnauba wax and beeswax**: For both the properties (water vapour barrier and grease barrier) I am using **mixture of carnauba wax and beeswax** in different proportions for checking the properties at different proportions. Mixing will be done on the oven by melting these wax at their melting temperature (MP for beeswax and carnauba wax are 61°C and 80°C respectively.). Then the coating will be done by the spray coating also can be done by dispersion coating but spray coating will give us a proper setting of material on substrate of both type having regular or irregular surface. Then cool it at different temperature for various results. The coating will cover the following aspect of the packaging for food :
 - a) The packaging is compostable (industrial or home compostable.)
 - b) The packaging is food borne.

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- c) The packaging is use for the short term applications.
- d) The packaging can sustain the temperature up to 60°C for hot food.

The previous work done on the paper material which have flat surface and have the following results :

Beeswax to Carnauba Wax	Annealing Temperature	Contact Angle	Sliding Angle	
	(°C)	(°)	(°)	
7:3	25	146.5 ± 1.8	<u>~</u>	
NA	60	159.7 ± 1.3	5.3	
5:5	25	148.7 ± 4.1	1. 6.	
621	60	167.1 ± 3.6	5.6	
3:7	25	139.6 ± 2.9	138.64	
5 1	60	159.0 ± 3.5	>10	

 Table 3: Water contact angles and sliding angles of various wax mixture latex coated paper.[3]

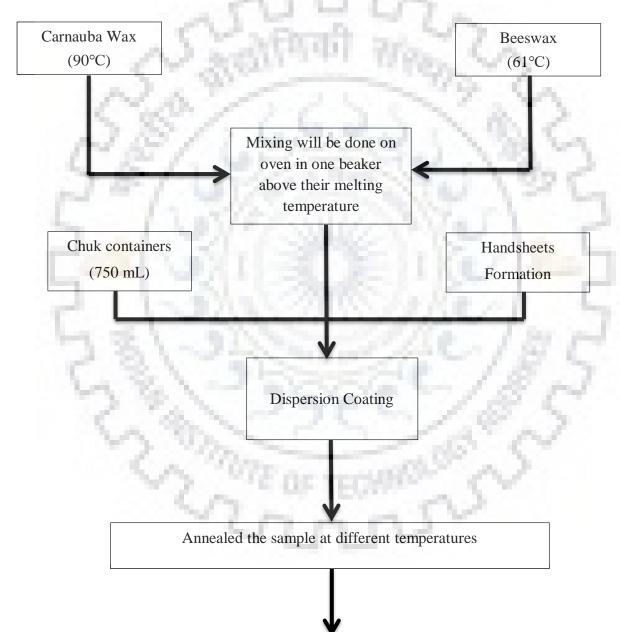
2.2. Chitosan–caseinate bilayer coatings: Sodium caseinate (SCAS) is commercially available and can easily form cohesive films from aqueous solutions because of its random coil nature and its ability to form extensive intermolecular hydrogen, electrostatic, and hydrophobic bonds. Films made of SCAS are colourless, tasteless, odourless, transparent, flexible, highly impermeable to oil and oxygen and resistant to thermal denaturation. But it seems the caseinate having lower strength and stiffness so it is not that much useful for the food application. But it can be used with multilayer structure having good structural properties and can be used with the food application. Thus the chitosan is used with the caseinate to impart some structural property also it will provide the good oxygen barrier and grease barrier coating. So we can get our required properties of water vapour barrier and grease barrier properties. There can be three steps of coating methods. Single-layer method in which caseinate solution was applied to paper by coater machine. Bilayer coatings were prepared by application of chitosan solution either on wet caseinate-coated paper (bilayer coatings 1) or on dry caseinate-coated paper (bilayer coatings 2).[4]

2.3. Bilayer-coated with chitosan and beeswax : It was observed that as the concentration of chitosan solution increased from 1.0 to 3.0 wt%, its water vapour transport rate (WVTR) decreased from 171.6 to 52.8 g/m²/d. Chitosan has good grease and oxygen barrier properties but has susceptible to the water vapour. So to improve this property of the substrate we will do beeswax coating over it to reduce the water vapour transmission that also increase the grease barrier properties.[5]

These are some following approach to reduce the plastic or polymer waste and use of the sustainable materials which will reduce the bad impact on the environment. Some other biopolymer have the same properties to barrier these properties. By this we can replace the non-degradable polymers.



This is the work methodology of the above procedure discussed in innovative approach. The above three methods are used to impart the different properties on the handsheets or packaging applications. But from the above three method, we choose, which is easy to process at college level and also at industrial level, the first one.



3.1. Mixture of carnauba wax and beeswax

Sample prepared



4.1. Sample preparation:

Figure 1: Waxes used for coating.

First we have 750 mL CHUK container made by YASH PAPER LIMITED as shown below in the figure. This container was cut into 13*14 cm² area. Then I checked the GSM of the paperboard using weighing balance. Then the paperboard was coated using dispersion rod and hot plate. Sample kept on the hot plate to keep it hot and then wax in different ratio poured on to the paperboard then using dispersion rod it was dispersed onto the paperboard. Then the sample was prepared. The material for coating was used beeswax and carnauba wax. After preparing the sample we have done rest characterization which was useful for the confirmation of the results. This is the flow chart of the sample preparation.



Figure 2: Sample preparation.

4.2. Field emission scanning electron microscope

FE-SEM is, refer to field emission electron microscope, used to observe material morphology up to nano scale on the surface of the material.(10)

Principle:

A FESEM is microscope, as the name suggest, that works with electrons unlike light microscope which works with light. These electrons are emitted from a field emission source. These electrons scan the materials and gives a visual report in display. The purpose of FESEM is to see topographic details on the surface, which are very small in size, that may be as small as 1 nanometer. As above said, Electrons are emitted from a emission source, by heating it using current, and high electrical field gradient accelerate these electrons. In high

vacuum column the primary electrons are focused and electronic lenses deflect these electrons to produce a narrow beam and that narrow beam bombards the object. From that secondary electrons are emitted from the bombard spots on the object. Surface structure of the object decides the angle and velocity of secondary electrons. These secondary electrons are detected and produces an electronic signal. These elect6ronic signals are being amplified and transformed to a scan-image to the monitor.



Figure 3: Field Emission Scanning Electron Microscopy(FESEM).(11)

Sample Preparation:

To observe the SEM images the sample should be conductive in nature, if it is not make it conductive using gold, platinum or gold-palladium coating. The coating of very thin layer of about 1.5-3 nm), that which does not affect the sample. Further on, the sample must be sustainable at high vacuum and should not affect the vacuum, by losing gasses, chemicals or water molecules.

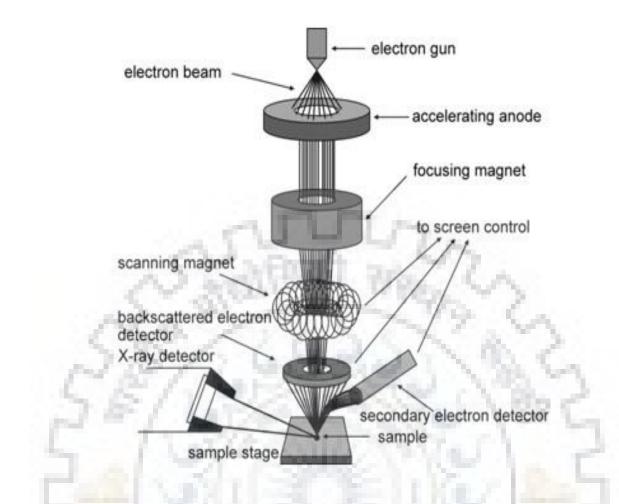


Figure 4: The set-up of a scanning electron microscope (12)

Source of electrons:

Generation of electrons in most of the standard electron microscopes by using heated tungsten filament up to a temperature of 2800°C by means of current. Sometimes crystal of lantanumhexaboride (LaB₆) is used to production of electrons kept on a tungsten filament. This modification gives higher density of electrons in the beam and gives a better resolution than conventional device. In FESEM microscope a "cold" source is employed. Tungsten needle with extremely thin and sharp diameter used as a cathode in front of a primary and secondary anode. The field applied between cathode and anode should be order of 0.5 to 30kV. The quality of image are better than the standard microscope about 1000 times smaller.

Image formation

When the primary electrons strike on the object, secondary electrons are emanated from the surface of that object having some velocity and the levels and the angles at the object surface are used to determine the velocity of that secondary electrons. These secondary electrons are attracted by the Corona, hit the fluorescing mirror or scintillator that produces photons. Illumination intensity and location of the mirror depending on the secondary electrons properties. The photons produced by the scintillator are amplified and transduced to a video signal that is fed to a cathode ray tube in synchrony with the scan movement of the electron beam. The contrast in the 'real time' image that appears on the screen reflects the structure on the surface of the object.

4.3. KIT test for grease barrier

Fluorochemical sizing agents are used to test the degree of repellency and for anti-wicking properties of paper and paperboard. To impart the organophobic and hydrophobic characteristics to the paper or paperboard fluorochemical may be used. These characteristics imparts through a reduction in the surface energy of the sheet.

By making 12 different solution for KIT test by using three reagent to make the solution. These three reagent are castor oil, toluene and n-heptane (according to TAPPI T559 test standard). These reagents are mixed in different ratio as mentioned in the table down there. With these reagents we made 12 solutions, with varying in viscosity and surface tension, from which the highest one was most aggressive.

2.09	the second		n-heptane,
KIT no.	Castor Oil, g	Toluene, mL	mL
1	969	0	0
2	872.1	50	50
3	775.2	100	100
4	678.3	150	150
5	581.4	200	200
6	484.5	250	250
7	387.6	300	300
8	290.7	350	350

9	193.8	400	400
10	96.9	450	450
11	0	500	500
12	0	550	550

Select a specimen to be tested, then put a drop of the intermediate solution the KIT no. and keep it for 15 sec then clean it with the cotton and examine the test area. If there is a darken area on the test side it means the test fail. If the specimen fails this first test, select an untested area on the same test specimen and repeat the test using the next lower numbered kit solution. Repeat until the highest numbered kit solution that rests on the surface for 15 s without causing failure is identified. If the specimen passes this first test, repeat the test on an untested area using a higher numbered kit solution. Repeat until the highest numbered kit solution that rests on the surface for 15 s without rates area using a higher numbered kit solution. Repeat until the highest numbered kit solution is the kit solution that does not cause failure is identified. The number of this kit solution is the kit rating for the specimen.

4.4. COBB Test

As we know the water absorbency has the great factor in packaging application mostly in paper and paperboard packaging. This test is used to determine the water absorbency of the paperboard material in a set period of time. Here, we determine the absorption of water per unit area. The apparatus required for COBB test are one water absorption apparatus, having cross sectional area of 100 cm² with corresponding diameter 11.28 cm, metal roller is made with brass, stainless steel or corrosion resistant material, the third apparatus is graduated cylinder and the last one is weighing balance. First the sample put on the water absorption apparatus, fills it with 100 mL water using graduated cylinder then keep it for 30 minutes then after 30 minutes check the water absorbed by the paperboard using weighing machine.



Figure 5: COBB apparatus.

4.5. Water Vapour Transmission Rate (WVTR)

This test is done to determines the rate of transmission of the water from the paperboard, either it is coated or uncoated. The setup below in fig.6 shows the procedure to conduct the WVTR test. This setup was self-prepared under the supervision of Dr. Ruchir Priyadarshi.

First we took the weighing bottle, aluminium foil, paraffin tape and vacuum grease. Then using these apparatus we created a setup of WVTR with transmission area of 1257 mm² corresponding to 20 mm diameter. After that we placed our coated sample onto the bottle over transmission area and sealed it with vacuum grease around its diameter. Then took these bottles into the desiccator and took the reading in every 24 hours.

These pictures shows that the 1st one the bottle before the WVTR test start, the 2nd one for during the WVTR test when placed it to desiccator vacuum free and the 3rd one for after the completion of WVTR test.

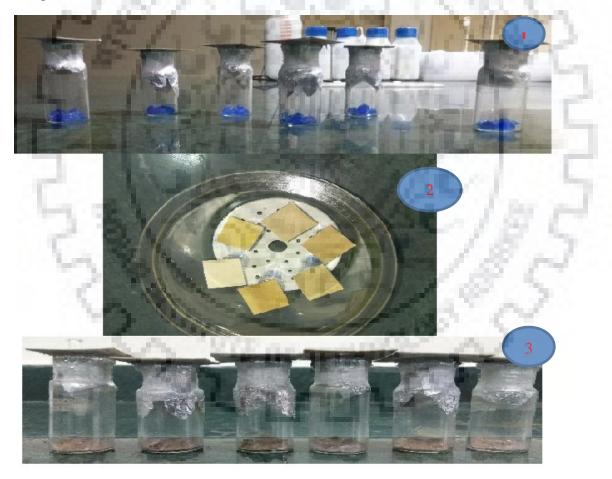


Figure 6: Water Vapour Transmission Rate setup.

4.6. Tensile Test

Tensile test is done by horizontal tensile testing machine.

Tensile strength is defined the maximum force required to rupture the specimen. This strength is calculated in force per unit width of the specimen.

Breaking length is defined by the maximum length up to which specimen would not break, while tensile force applied on it. It would break due to tensile force when it is beyond its breaking limit. The unit of tensile strength is measure in kilometre.

Here, we kept samples between two clamping jaws for gripping the specimen. Then set rate of separation of the clamping jaws, which provide tensile force to the specimen. When specimen reaches to its breaking limit it breaks. And we get the required values for the tensile test .



Figure 7: Horizontal Tensile Testing Machine.

5.1. Failures during the project

Firstly we tried coating with spray coated using melt waxes but during spray it was cooled and blocks the path for spray means the coating was not done with the spray coater.

Then we tried coating on sample with hand using cotton but the coating is not uniform cause in some places it showed a gap and also not get uniform coating on the paperboard and tried with coater rod without using hot plate, by this also the waxes cooled very fast and stick to the coater rod. These are some failure samples given below:



Figure 8: Failed sample after using different coating technique.

The table below shows the coating on the sample but the GSM of the coating is very high which affect the cost to the company. After testing was done on these samples which give good KIT value also COBB value in comparison to the uncoated sample. Then the coating GSM was reduced with increasing temperature variable. It also reduces the thickness of the coating which is economic to the industry. After increasing temperature of the hot plate the GSM was reduced to near about 30 GSM from 94 GSM with the same results.

SAMPLE	BEESWAX	BEESWAX : CARNAUBA WAX (7:3)	BEESWAX : CARNAUBA WAX (3:7)	CARNAUBA WAX
Coating Thickness (µm)	15	22.5	15	15
Coating GSM	91.15	104.65	81.30	89.60
KIT TEST	523	लिली क	2	
KIT value	10	12	12	
COBB 30 min	8 / 5 6	a. 10, 10, au	$1 < \infty$. C.
\mathbf{W}_1	8.79	8.15	8.20	7.96
W ₂	8.90	8.63	8.75	8.54
Difference	0.10	0.48	0.55	0.58
Water absorbed g/m ²	10	48	55	58

Table 5: KIT value and COBB value of failed samples.

From these results, by increasing coating GSM the COBB value decrease to very low level. The COBB value for uncoated paperboard is about 117 g/m², but when we increase the coat weight the COBB value is about 11 g/m². Means the coat weight on paperboard is inversely proportional water absorbed by the paperboard. When we reduce the coating weight it is around 25 GSM the absorbed value of the sample is around 62 g/m².

5.2. Morphology of the coated sample

Sample-1: This sample is made by coating of beeswax on the bagasse moulded paperboard. Firstly, the beeswax material is heated to above its melting point then the liquid form of the beeswax is dispersed on to the paperboard by pouring on it and then dispersed by rod. Here we get the GSM of the coated material around 22.65. The FE-SEM images show that the beeswax is almost uniformly distributed above the sample.

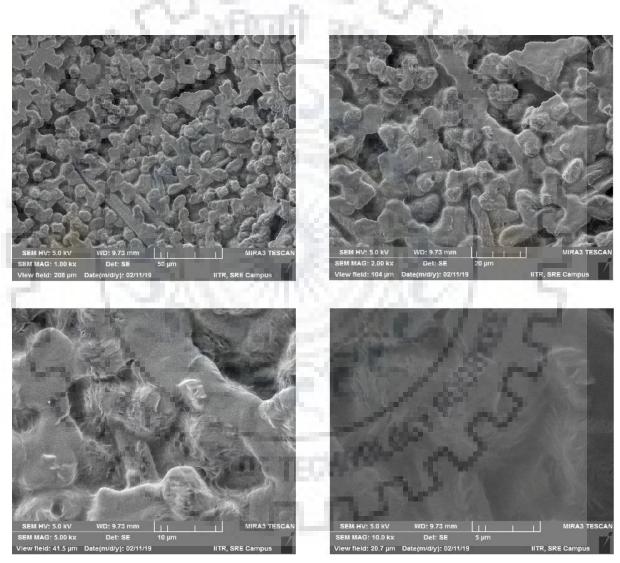


Figure 9: FE-SEM images of sample-1.

Sample-2: This sample is made by coating of mixture of beeswax and carnauba wax, the percentage of beeswax is 70% and carnauba wax is 30% in the total mixture of coated material, on moulded paperboard using dispersion coating. Here the GSM of the coating material is around 45.92. Here, from the image, we can say that the material is not distributed uniformly. This accumulation happens because the two different waxes has different waxes has different temperature, due to which one wax, carnauba wax, allows itself solidify faster than the other, which promotes shrinkage of the particles.



Figure 10: FE-SEM images of sample-2.

Sample-3: This sample is made by coating of mixture of beeswax and carnauba wax, the percentage of beeswax is 50% and carnauba wax is 50% in the total mixture of coated material (In the same ratio), on moulded paperboard using dispersion coating. Here the GSM of the coating material is around 22.24. Here, from the image, we can say that the material is not distributed uniformly.

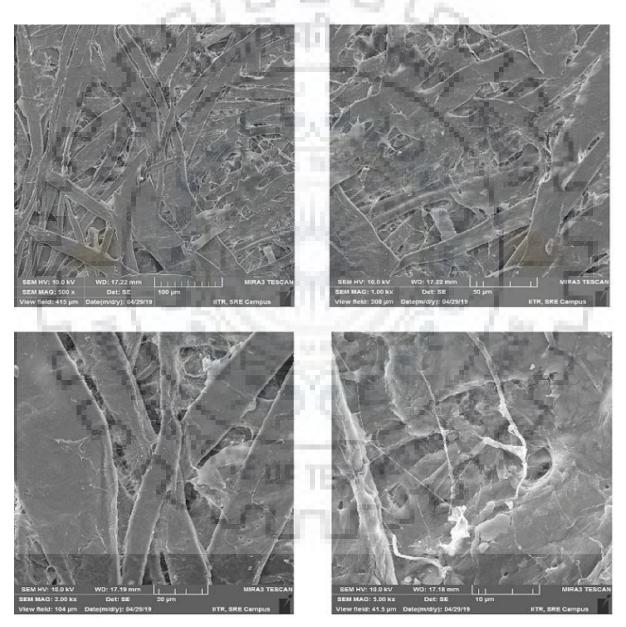


Figure 11: FE-SEM images of sample-3.

Sample-4: This sample is made by coating of mixture of beeswax and carnauba wax, the percentage of beeswax is 30% and carnauba wax is 70% in the total mixture of coated material, on moulded paperboard using dispersion coating. Here the GSM of the coating material is around 34.61. As the ratio of the carnauba wax increase in the solution the accumulated particle size increase. Here, from the image, we can say that the material is not distributed uniformly. As discussed in the sample 2 the accumulation of particle occur due to the melting point difference of the waxes. Here the ratio of carnauba wax is higher than the beeswax comparative to the above sample-2, so here the accumulated particle size is little bigger than the sample-2 particle size.

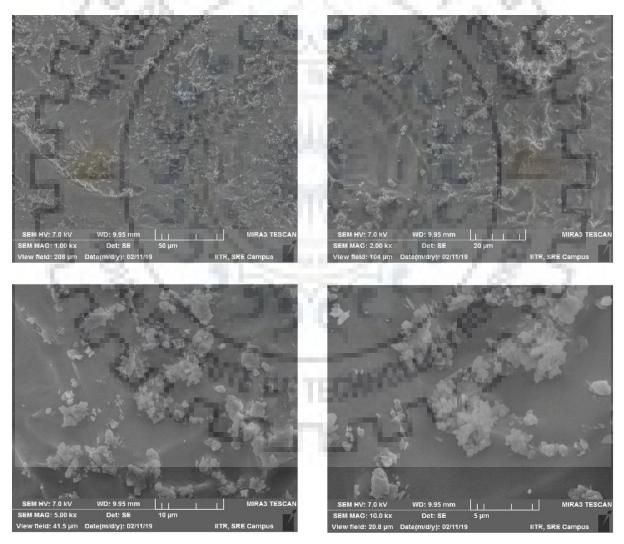


Figure 12: FE-SEM images of sample-4.

Sample-5: This sample is made by coating of carnauba wax only on moulded paperboard using dispersion coating. Here the GSM of coating is about 23.67. Firstly the carnauba wax is heated to its melting temperature, then pour onto the sample then dispersed it on the substrate using rod.

The FE-SEM images show that the beeswax is almost uniformly distributed above the sample. The FE-SEM images of this sample are taken at different magnification which is given below:

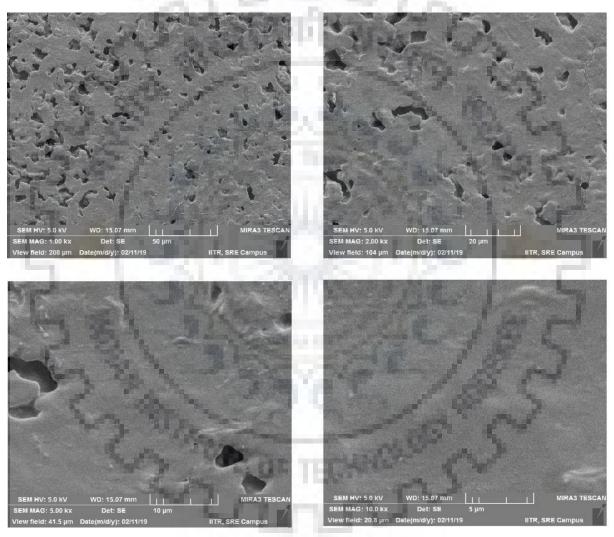


Figure 13: FE-SEM images of sample-5.

5.3. FT-IR Analysis of the data

Both the waxes (Beeswax and Carnauba wax) have composition of same type elements like beeswax consists of esters of fatty acids and long-chain alcohol, similarly the carnauba wax consist of aliphatic esters, diesters of 4-hydroxycinnamic acid (21.0 wt%), ω -hydroxycarboxylic acids, and fatty alcohols. So the plot of both the waxes is almost same. One or two peaks of both differentiate the waxes using their peak value.

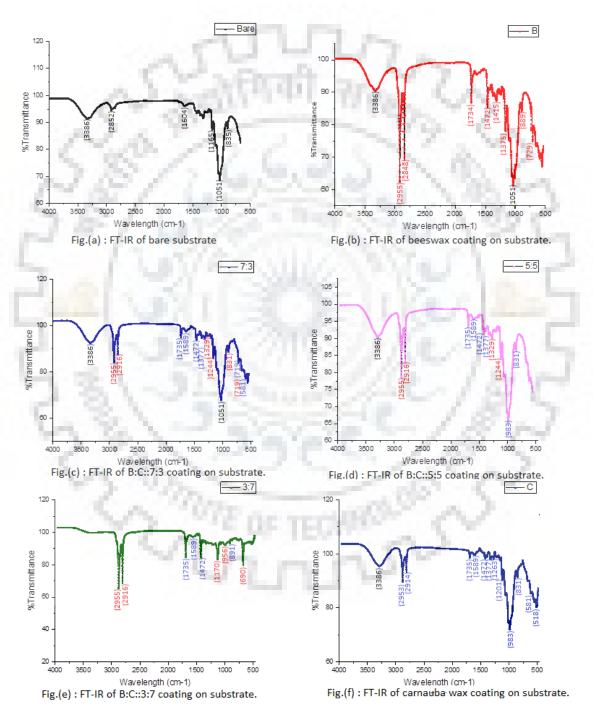


Figure 14: FT-IR of all the samples with different wax ratio.

From the above figure, data taken from FTIR machine and graph plotted in OriginLab software, fig.(a) shows the FTIR data of the bare substrate means uncoated sample of bagasse molded paperboard. In all the plotted graphs the peaks of bagasse are denoted using black numbers with its wavelength in the small bracket. Some of the peaks of bagasse and waxes are showing the same results.

Fig.(b) shows the FTIR graph of beeswax in which some peaks of beeswax and some peaks bagasse. Fig.(c), fig.(d) and fig.(e) show the plots of mixture of beeswax and carnauba wax in the ratio of 7:3, 5:5 and 3:7 respectively. Fig.(f) shows that the FTIR plot of the carnauba wax coating on the bagasse substrate.

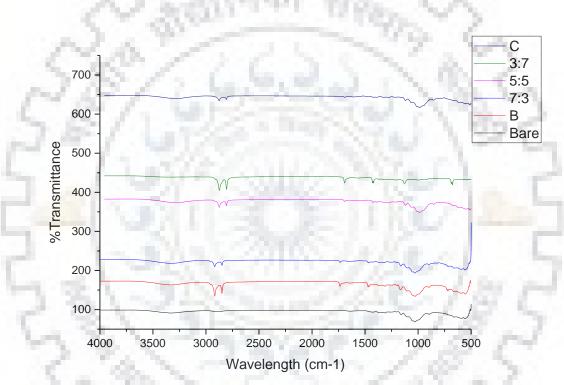


Figure 15: Comparison analysis of FT-IR data.

The conclusion from the above plotted graph is that there is some peaks which differentiate the coating materials on the substrate like peaks at 690 cm⁻¹ wavelength shows the material is beeswax which peak is not present in carnauba wax and bagasse plot. Also the bagasse plot shows that the peaks at 3386 cm⁻¹ and 1051 cm⁻¹ which shows in every plot. And the rest values are matched with the ref.

5.4. COBB and KIT test

As there the KIT and the COBB test is done on the sample with least achieved coating GSM. The KIT test method procedure was done as mentioned in materials and method chapter. By mixing of four different reagent 12 different solutions were prepared using which we have done KIT test.

			the second second		
SAMPLE	SAMPLE BEESWAX		B:C	B:C	CARNAUBA WAX
	1.1	7:3	5:5	3:7	
W1	1.782	1.819	1.818	1.8044	1.812
W2	1.913	2.044	1.966	1.974	1.969
DIFFERENCE	0.131	0.225	0.148	0.1696	0.157
GSM-1	26.73	45.92	30.2	34.61	32.04
6 8	1.1				Sec. 3.
W3	1.806	1.753	1.828	1.792	1.789
W4	1.917	2.218	1.937	1.965	1.905
DIFFERENCE	0.111	0.465	0.109	0.173	0.116
GSM-2	22.65	94.89	22.24	35.3	23.67
Contraction of the local division of the loc					
THICKNESS		1.00			1
Τ1 (μ)	595	595	595	595	595
Τ2 (μ)	600	605	612.5	605	597.5
Ta Sa	5	10	17.5	10	2.5
100 C				- / -	S. And
Τ1 (μ)	595	595	595	595	595
Τ2 (μ)	600	615	600	607.5	600
Ть	5	20	5	12.5	5
	\sim			$\sim \sim$	
KIT Test	6.54			1. 1	
KIT Value	10	11	11	10	11
COBB Test					
Wa	22.65	45.92	22.24	34.61	23.67
Wb	23.27	46.4	22.98	35.20	24.25
Difference	0.62	0.48	0.75	0.59	0.58
g/m²	62	48	75	59	58

Table 6: Results of KIT test and COBB test

KIT value of the uncoated sample was 2. When we see the KIT value of the coated sample, it gives the good result. As it seen in the table the KIT value of the beeswax coated sample is 10. Similarly others samples have KIT value is 11, 11, 10 and 11 of beeswax with the ratio of carnauba wax is 7:3, 5:5, 3:7 and only carnauba wax respectively.

Coming to COBB test the values of COBB shows the absorption of water into the sheet per unit area. The COBB value of the uncoated sample is 117 g/m2. The value of coated samples is reduced more than 35% of the uncoated sample. The COBB value of the coated sample is shown above table.

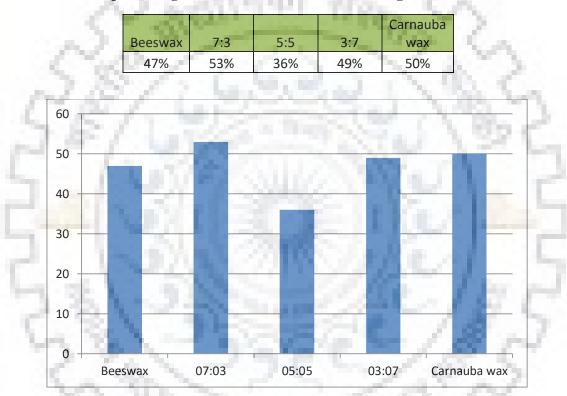


Table 7: Reduced percentage of water absorbed after coating.

Figure 16: Graph of reduced percentage of water absorption after coating.

The above graph shows the how much percentage of water absorption reduce after coating onto the uncoated samples. The above data shows a marginal reduction in the water absorption into the coated samples in comparison to the uncoated sample.

5.5. WVTR characterization

After prepare sample of different coating material ratio the main objective of the project is to check the WVTR (Water Vapor Transmission Rate). The setup for WVTR was prepared using a desiccator, silica gel, weighing bottle (25mm*60mm) and water. First the weighing bottles were made isolated from the environment means sealed from all the sides except leaving Transmission area of 1257mm²(with 20mm diameter). Then placed the 5 coated and 1 uncoated molded paperboard over that transmission area and placed these weighing bottles into the desiccator with 99% RH and 27°C temperature. The increased weight of weighing bottles (absorption of water vapor) was measured using weighing machine in every 24 hours.

Sample/Weight	OHrs	24Hrs	48Hrs	72Hrs	96Hrs
10 mg	g	g	g	g	g
Bare	13.797	13.99	14.117	14.251	14.337
Beeswax	14.251	14.325	14.409	14.451	14.489
07:03	14.623	14.667	14.731	14.74	14.744
05:05	14.444	14.532	14.623	14.662	14.705
03:07	14.271	14.353	14.448	14.454	14.502
Carnauba Wax	14.652	14.763	14.861	14.926	15.012
1 1 2 10					
Difference in weight					-
Bare		0.193	0.127	0.134	0.086
Beeswax		0.074	0.084	0.042	0.038
07:03		0.044	0.064	0.009	0.004
05:05		0.088	0.091	0.039	0.043
03:07		0.082	0.095	0.006	0.048
Carnauba Wax		0.111	0.098	0.065	0.086
C. S. V				1.58	
WVTR value per day		24Hrs	48Hrs	72Hrs	96Hrs
Bare		91.356	60.115	63.429	40.708
Beeswax		35.322	40.095	20.048	18.138
07:03		21.527	31.313	4.403	1.957
05:05	5 (BC 11	42.005	43.437	18.616	20.525
03:07		39.467	45.724	2.888	23.103
Carnauba Wax		52.983	46.778	31.026	41.050
% Reduce in WVTR in					
comparison to bare sample		24Hrs	48Hrs	72Hrs	96Hrs
Beeswax		61	33	68	55
07:03		76	48	93	95
05:05		54	28	71	50
03:07		57	24	95	43
Carnauba Wax		42	22	51	-1

Table 8: Day to day analysis of WVTR of coated and uncoated samples.

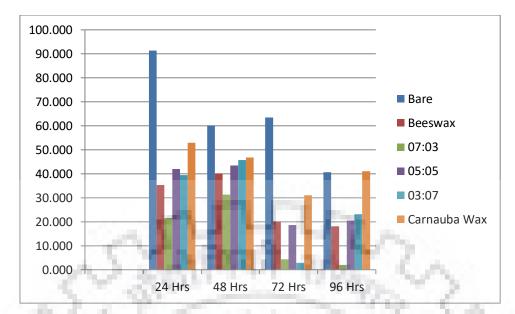


Figure 17; Day to day WVTR analysis of coated and uncoated samples.

By checking the weight increase per day were tabulated in the table using which WVTR value can be calculated. The formula for WVTR value is:

WVTR =
$$\frac{\text{Sample Weight Change * Thickness of Film}}{\text{Transmission Area * Time}} \left(\frac{\text{g*mm}}{\text{m}^{2*} \text{ day}}\right)$$

Using above formula, the value of WVTR has been calculated. From the above tabulated value the value of WVTR have been reduced maximum up to 76% of the sample having beeswax and carnauba wax ratio is 7:3 with respect to the bare sample on the first day of the WVTR experiment. From the other also get the good results like beeswax, beeswax and carnauba wax in the ratio 3:7 and 5:5 and only carnauba wax reduces a marginal WVTR values which reduces around more than 40% in every sample on its first day of the experiment.

The marginal difference between the sample 2 (beeswax : carnauba wax :: 7:3) and other sample is due to the coating GSM and coating thickness between this sample and other sample, which creates that much difference in WVTR value. The WVTR test was conducted for the 5 days with four values of increased weight, after that the value of sample get constant.

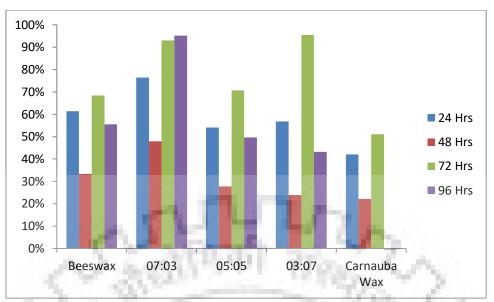


Figure 18: Percentage reduce in WVTR in comparison to bare sample.

5.6. Tensile Test results

Tensile test results show no that remarkable effect after coating. The results of the coated samples and uncoated sample have not that much difference. The variation of results in the sample after coating may be cause of the sample defects. In some places the porosity might be high or the coating is not proper. So the effect of coating of these waxes on paperboard is not much effective for getting the tensile properties.

Sample	Uncoated Sample	Beeswax	B:C 07:03	B:C 05:05	B:C 03:07	Carnauba Wax
Tensile Strength Index	44.65	46.38	47.08	41.86	46.39	43.45
(Nm/g)	1					
Tension Length	4.56	4.73	4.8	4.27	4.73	4.43
(km)						

Table 9: Result of tensile testing (Tensile Index and Tension Length).

6. CONCLUSION

As the coating is done on the paperboard with the beeswax and carnauba wax to get the good water vapour properties and grease proof property. The beeswax and carnauba wax was mixed with each other at different ratio to check the properties. Coating with only beeswax and only carnauba wax showed, in FE-SEM images, uniform coating on the samples. But when we go through the mixed coated structure the coating was not get that much uniform, it is because the coating materials have different melting points which give some particles type structure on the coated samples. With FT-IR analysis we get the peaks of both carnauba wax and beeswax also bagasse peaks, matched with the literature part. The FT-IR confirms the materials combination in the coating. The KIT value of all the coated samples is greater than 10 while the KIT value of uncoated sample is 2. Going to the COBB test the best result comes from this test from sample-2 (coated with beeswax and carnauba wax with 7:3 ratio) which reduces the 53% (from $117g/m^2$ to 48 g/m²) water absorbance capacity of the paperboard. COBB also culminated that on increasing coating weight the value of absorbance decrease. Also from the WVTR results the water transmission rate decreases to 76% from the sample-2. But there is not much effects in the tensile properties of samples after coating.



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