

A

DISSERTATION REPORT

ON

**Investigation of the effect of additives on the dewatering
behaviour of highly refined cellulose pulp**

Submitted in partial fulfillment of the requirement for the award of degree of

MASTER OF TECHNOLOGY

By

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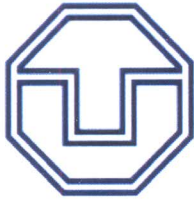
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CANDIDATE'S DECLARATION

I hereby declare that the work which is being represented in this dissertation report entitled as **“Investigating the effect of additives on the dewatering behaviour of highly refined cellulose pulps”** in the partial fulfilment of the requirement for the award of degree of Master of Technology in Packaging Technology and submitted in the Department of Paper technology, IIT Roorkee (SRE-Campus) is a record of my own work carried out under supervision of Associate Professor Dr. Chhaya Sharma and Prof. Dr.-Ing. André Wagenführ, Dipl.-Ing. René Kleinert.

The matter embodied in this project report has not been submitted by me for the award of any other degree of this or any other Institute. I have given the due credit whichever I have used in this report.

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Abstract

TU Dresden developed 100 % fiber-based biogenic materials which show superior properties being able to substitute fossil-based polymers in different lightweight applications. Thus, the developed cellulose films have a great potential to exploit new markets such as packaging that requires high strength properties as well as barrier properties. For further application-oriented development it is of importance to improve the existing subsequent process like dewatering especially in terms of process time and energy consumption.

The aim of the envisaged thesis is to characterize and to investigate the effect of wet end additives on the dewatering behavior of highly refined cellulose pulps (FibCell). Zeta potential was considered as a basic factor to study the dewatering behavior on addition of different additives. Effects of these additives were evaluated. A composition with reasonable trade between the properties is found using an Optimizer tool Modde12.1.



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1 INTRODUCTION

1.1 Packaging

The world's population has more than doubled over the past 50 years from just over three billion in 1960 to almost seven billion today. With this population growth, we have also seen a massive increase in the demand and production of goods and services. The size of the global economy has grown from a mere 1.35 trillion dollars in 1960 to over 60 trillion now. This massive growth in economy leads to a necessity to deliver products over the long demographic regions [1].

Packaging is the technology which exists for the purpose to deliver products to final consumer in safe and sound condition. Certain features in packaging are required to be included according to the legislative and regulatory requirements as well as environmental condition which varies with demography of region where the product is needed to be delivered. Packaging should designed in such a way that it could withstand the different conditions to which it is subjected during transportation and handling [2].

Various types of materials are used in packaging (Plastics, Metals, wood, Glass and Cardboards) depending upon the product specification and usage. Most commonly used packaging material are petroleum based polymers because of there superior properties and efficient economic aspects. The annual production of Plastics has inncreased from 1.5 million tonnes in year 1950 to 299 million tonnes in 2013[3]. But they comes from non-renewable sources and require unknown amount of time for there degradation when exposed to UV radiation of sunlight in marine. It is due to the oxidative properties of atmosphere and hydrolytic properties of seawater, these polymers become embrittled and break into individual polymer molecule [4].

Material	Plastic Bottles	Plastic Bags	Tin Can	Glass Bottles
Decomposition Time (years)	70-450	500-1000	50	1-2 million

Table 1: Decomposition of different packaging materials[5]

Due to long amount of time required for decomposition of plastics a huge concern arises related to waste management. Packaging waste accounted for 31.6% of the total municipal solid waste (MSW) in 2003 in the USA, 25% of the MSW in 2005 in Europe, and 10% of the MSW in 2004 in Australia [6].

Every year 8 million tonnes of plastic is dumped in our oceans and they are very harmful for ecosystem [7]. Some of the plastic have been proved carcinogenic. So, we are looking for an alternative material which could provide similar properties as of plastics and could be degrade in environment without causing much damage.

1.2 Cellulose

Cellulose is most abundant organic polymer on this planet with an annual production of 1.5×10^{12} tonnes [8, 9]. It is major constituent of cell wall of plants. Cellulose can be obtained from by-products of crops such as straws of rye, wheat, rice, oats and sugarcane, corn stalks, sorghum bagasse and from wood as well. Aside from being building block of plant cell, cellulose has many other uses. Cellulose can be used to produce paper film, explosives and plastics depends on how it is treated.

Material	Cellulose content
cotton	95%
flax	80%
jute	60-70%
wood	40-50%

Table 2: Cellulose content in different materials

Cellulose can be simply described as Carbohydrate. Carbohydrates are organic compound made up of carbon, hydrogen and oxygen which acts as immediate energy source for living entities. Plants make glucose to use it for energy or to store as starch for later use. A plant uses glucose to make cellulose when it links many simple units of glucose together to form long chains. Cellulose is linear polysaccharide of anhydro-glycose consisting of several thousands of β -1,4-glycosidic linkages. Due to its rigid structure Cellulose is resistant to degradation and chemical attacks [10].

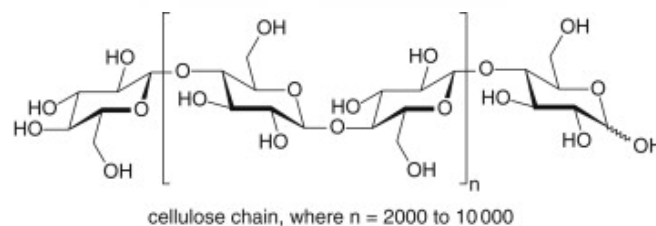


Figure 1: Schematic structure of cellulose.

1.3 Cellulose Properties

In its pure state cellulose is white in color. Cellulose is non-toxic, odourless as well as tasteless. It has a melting point of 467 °C. Generally It has very low conductivity. Cellulose is biodegradable. Cellulose fibers are hydrophilic in nature due to the presence -OH group at their surface. Cellulose is known to be insoluble in water due to the large amount of strong hydrogen bonds which suggests it will be soluble in a polymer that can destroy this interchain hydrogen bonding in cellulose. At extreme pH it can be dissolved in water if a cosolute of intermediate polarity is added [11].

1.4 Objective

Petroleum based packaging materials are harmful for our ecosystem as well as cause certain impact to human health if used for long duration. TU Dresden developed 100 % fiber films with highly refined cellulose that shows superior strength and barrier properties which are very important for packaging aspects. With the superior properties of fiber film, it may be able to substitute fossil-based polymers in different applications. For industry scale production there is a need to improve dewatering process especially in terms of process time. The aim of the envisaged thesis is to characterize and to investigate the effect of additives on various properties and dewatering behavior of highly refined cellulose pulps.

2 LITERATURE REVIEW

2.1 *FibCell*

The Professorship of Wood Technology and Fiber Materials Technology at TU Dresden is aiming at exploring new ways for potential application of cellulose specially in light weight material. In collaboration with a mill for specialty papers a highly fibrillated cellulose pulp called FibCell was developed.

2.2 *FibCell Properties*

FibCell has a Young's modulus of 11.4 GPa and tensile strength of 89 MPa which are superior to most of the polymers. Additionally it has a dense structure which results in excellent barrier properties against grease and oxygen. FibCell have very good electrical insulation. A very low density of 1.54 g/cm^3 serves a complementary roll in making it suitable for high strength and low weight application.

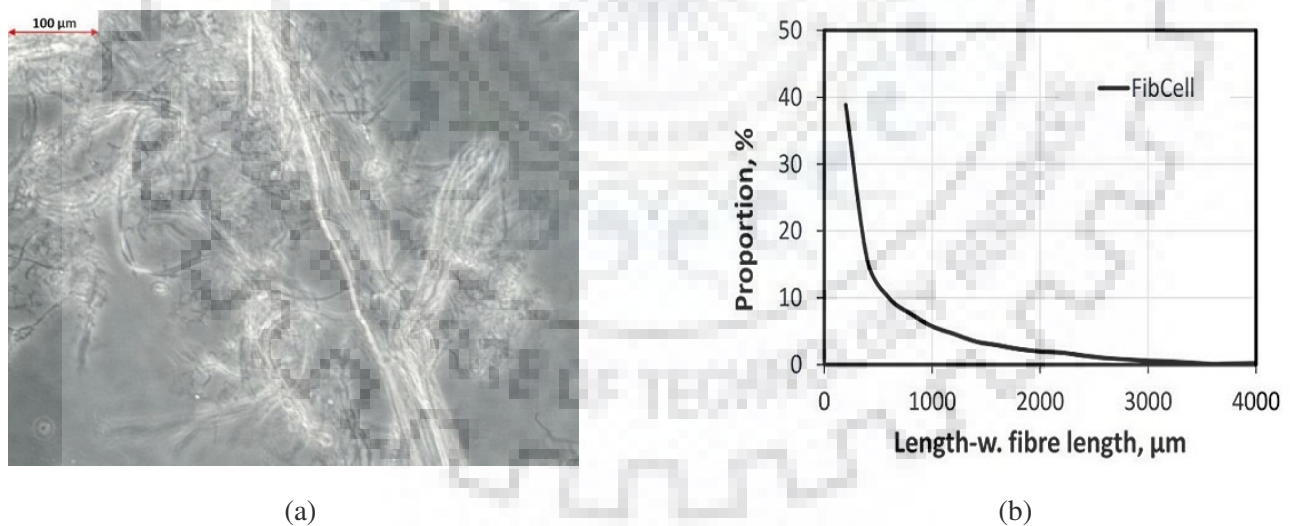


Figure 2: (a) Microscopy picture of FibCell pulps [13], (b) Length-w. fibre length distribution of FibCell pulp [14]

In order to obtain high strength, it was necessary to provide a high content of fibrils so as to provide a high specific surface for the formation of hydrogen bonds in dehydrated product. Pulp used for experimentation purpose was a specially treated NBSK (Nordic Bleached Softwood Kraft) pulp. Conical

refiners were used to beat the pulp to a Schopper Riegler value over 85 SR or to a CSF (Canadian Standard Freeness) level lower than 100 ml. The difference between this pulp and MicroFibrillated Cellulose or NanoCrystalline Cellulose is due to the presence of residual fibers and fiber fragments but latter have less or no longer fibers and particles [12].

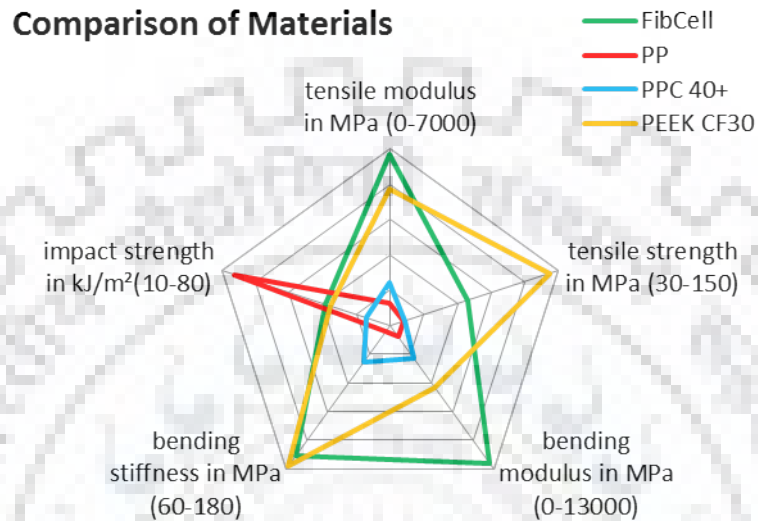


Figure 3: Comparison of materials used in lightweight construction; FibCell - 100%, PP - Polypropylene, PPC - PP reinforced with 40% fibers from paper residues, PEEK - polyether ether ketone reinforced with 30% carbon fibers [13]

Fig.3 shows a comparison between the result of strength properties obtained for FibCell as compared for other materials. It can be observed that tensile modulus and bending modulus as well as tensile strength and bending stiffness have a considerably higher values for FibCell as compared to those of pure plastics and commercially available NFRCs. The same applies to the comparison with CFRCs, such as PEEK CF30 while tensile strength was found to be lower but in the same range like aluminum [13].

Due to the large amount of fibrils and fines as well as long fiber and fiber fragments (see fig.2a) the pulp requires exceptionally longer duration in dewatering process. This makes the material unjustified for Industrial use although FibCell have a density slightly higher than half of aluminum and 80 % lower than that of steel.

2.3 Zeta Potential

In papermaking systems a charge develops at solid-water interface due to dissociation of carboxyl and sulfonic acid groups on the fiber surface caused by adsorption of hemicellulose and dissolved lignin or various charged additives onto fiber and fines surface.

The surface charge is anionic in nature in papermaking. Its intensity depends on the type and surface density of charged group (e.g. carboxyl groups or sulfonic groups). The ions of opposite charge (counterions) are held to the adjacent surface by electrostatic force of attraction. In this region a rapid drop off of potential energy take place. Between tightly bound counterions adjacent to the surface (stern layer) and rest of solution there exists a hydrodynamic slip plane. The less ordered region where potential energy drops off at a slower rate as compared to stern layer until zero is called Gouy-Chapman Region [15].

The potential at the interface of stern layer and Gouy-Chapman Region is called Zeta potential. Surface charge measurements involve measuring zeta potential rather than actual surface charge. There is no overall charge on suspension. These potential determine the resistance of suspension to coagulation or flocculation.

Flocculation - The destabilization of a colloidal suspension caused due to bonding of the particles together with a long chain polymer.

Coagulation - The destabilization of a colloidal suspension caused due to bonding of the particles together with low molecular weight substances like salts [15].

2.4 Effects of Zeta potential

2.4.1 Dewatering

Flocculation of cellulose can be induced by charge neutralization and patch formation technique using different types and concentration of additives. Depending upon the type of salt addition aggegration and sedimentation of fibers occurs due to the compression of electrical double layer. The viscosity was found to be increased with increase in ionic strength of the suspension.

The dewatering ability of pulp suspension is directly related to the degree of aggregation of fibers.

In order to remove water from fiber suspensions, channels for water flow through fiber network are needed. By addition of cationic additives or salts larger channels can be formed due to charge neutralization. That is, as larger flocs are formed there is an increase in lower-density areas between the flocs which provide paths for drainage [16, 17]. But when the amount of additive passes a particular limit dewatering ability again decreases as a result of reduction in the size of these drainage channels due to increase in electrostatic force of repulsion.

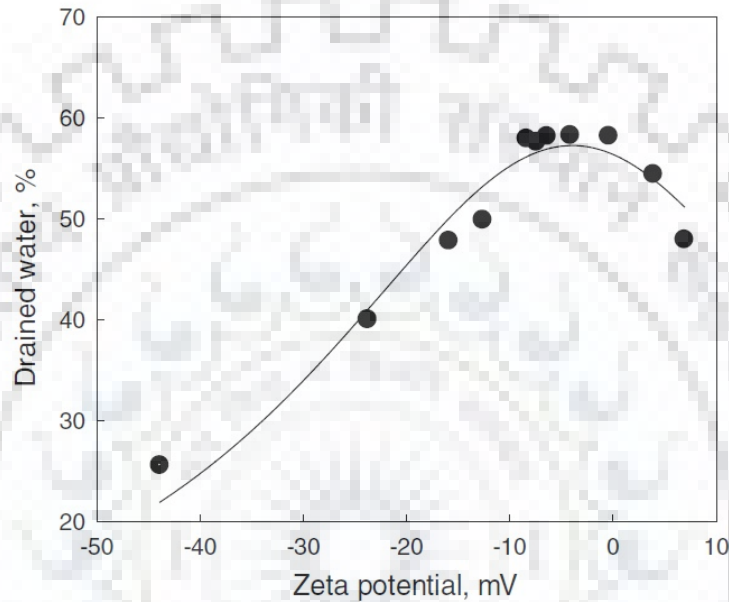


Figure 4: Relationship between zeta potential and derained water amount of CNF suspension with salt in 5 min [16]

The fig.4 shows the relationship of zeta potential with amount of water drained. It can be observed that as zeta potential approaches to zero (neutral charge point) by addition of salts the percentage of water drained increases. This indicates that dewatering ability can be increased by controlling zeta potential [16].

2.4.2 Viscosity

The viscosity of fiber suspension decreases with increase in strain which shows a typical shear thinning behavior. At low strain conditions the viscosity of additive or salt free suspension was found lower than that of on with added additive. The thickness on electric double layer decreased on salt or additive addition causes reduction in repulsive force. As a result the fibers can flocculate and it promotes a

stronger network structure of fiber suspension with higher viscosity. Bivalent salts showed higher effects on viscosity.

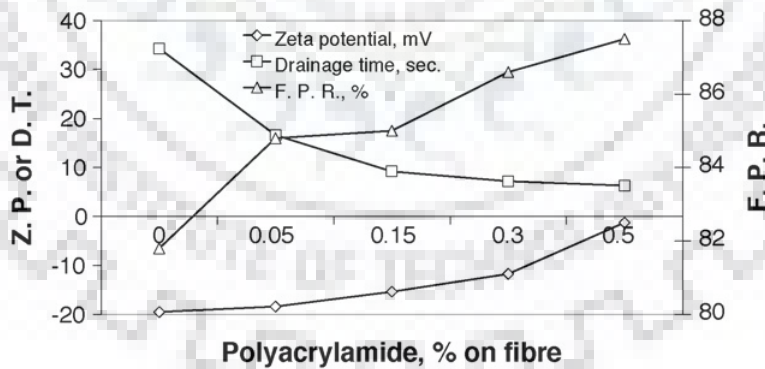
2.4.3 Retention

Zeta potential not only plays an important role in Coagulation/flocculation and and dewatering but it can also be used to improve the net retention of fines and additives. As zeta potential approaches to zero, conditions for dewatering and retention approaches to optimum [18]. When an additive is used to reduce zeta potential then the additive cause a complex bridge formation between fiber, fines and fillers. This leads to a complex strong network which as a result acts as a retention aid.

2.5 Additives

On the basis of literature survey three additives were studied for the purpose to achieve better dewatering and other properties as the showed very favourable results with other materials.

2.5.1 Polyacrylamide



Z. P. - Zeta Potential; D. T. - Drainage Time; F.P.R. - First Pass Retention.

Figure 5: Effect of polyacrylamide on zeta potential dewatering and retention of OCC pulp [18]

Fig 5 shows the effect of polyacrylamide on OCC pulp. Both drainage and first pass retention was increased with the addition of additive. As zeta potential approaches to zero the maximum percentage of improvement was observed.

Dose on O.D. pulp(%)	Improvement in drainage(%)	First pass Retention(%)
0.05	52	84.8
0.15	73	85
0.3	79	86.6
0.5	82	87.5

Table 3: Effects of using PAM in OCC

2.5.2 Cationic Starch

Normally with hydrogen bonding repulsive electrostatic forces exists between fiber-fiber and fiber-filler due to the presence of anionic charge. When cationic starch is added to fiber suspension besides hydrogen bonding, several new bonds are formed. This leads to a complex bridge formation, appears as flocculation effect.

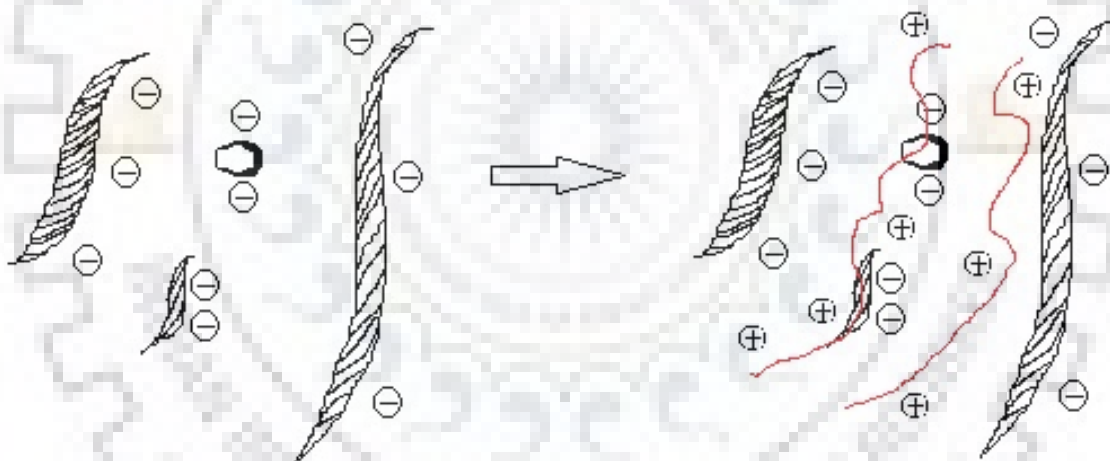


Figure 6: Graphical representation of interaction of starch with fibers, fines and fillers

The extra bonding caused due to addition of cationic starch also results in better mechanical properties (i.e. tensile strength, burst strength etc). Due to the large flocs formation cationic starch also helps in increasing drainage.

3 METHODOLOGY

3.1 Strategic plan

The fig.7 shows a flow diagram of the sequential steps used to perform experiments. Firstly the properties of pure FibCell without any additives are measured as a reference to be compared with the later values when chemicals will be mixed into the pulp suspension. The next step involves adding desired quantity of different retention and drainage aids to the pulp. Holding the pulp while stirring for a minimum time so that pulp can be homogenized and provide papermaking conditions. Dewatering time for the homogenized pulp is measured. Then, the impact of these retention aids on other properties are studied.

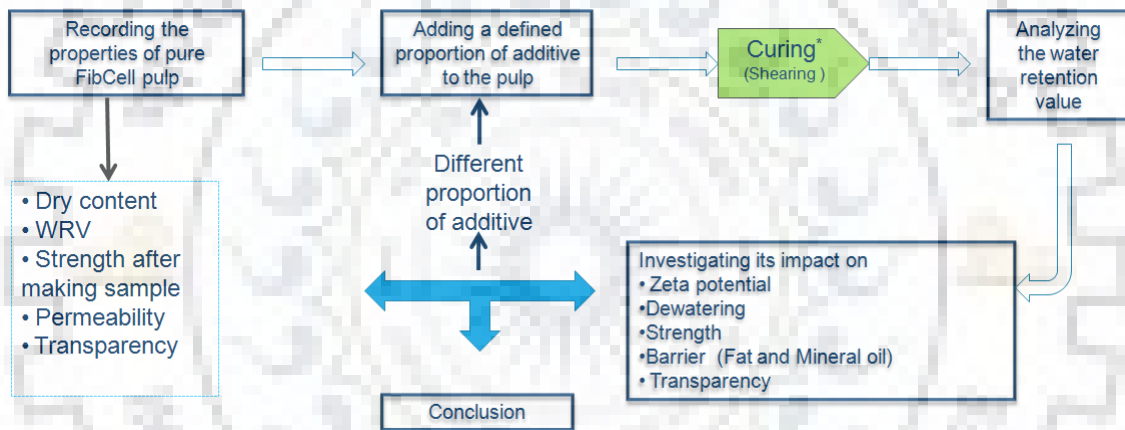


Figure 7: Process Flow Diagram

3.2 Selection of Additives

On the basis of literature review we observed that there is a direct co-relation between zeta potential and dewatering of fibers suspension. So, while selecting the additives as well as in determining their concentration used zeta potential was the main ground. Chemicals are selected on the basis of their capability to alter zeta potential. Exceptionally Calcium carbonate was used in order to create spaces between the fibers so the the water can rinse through the gap. As zeta potential approaches to zero it was previously found that dewatering was optimum. As at zero zeta potential the repulsive forces between fibers and fines reduced to minimum which cause subsequent flocs formation. Therefore, it

was taken the upper limit concentration of that chemical. The lower limit was taken as zero to get an idea of wider range. In order to determine the limits, experiments were conducted for determining variation in zeta potential with variation in concentration of following chemicals.

3.2.1 Cationic Polyacrylamide

cPAM used was supplied by Kurita Labufloc 11470. It was weakly cationic with medium average molecular weight.

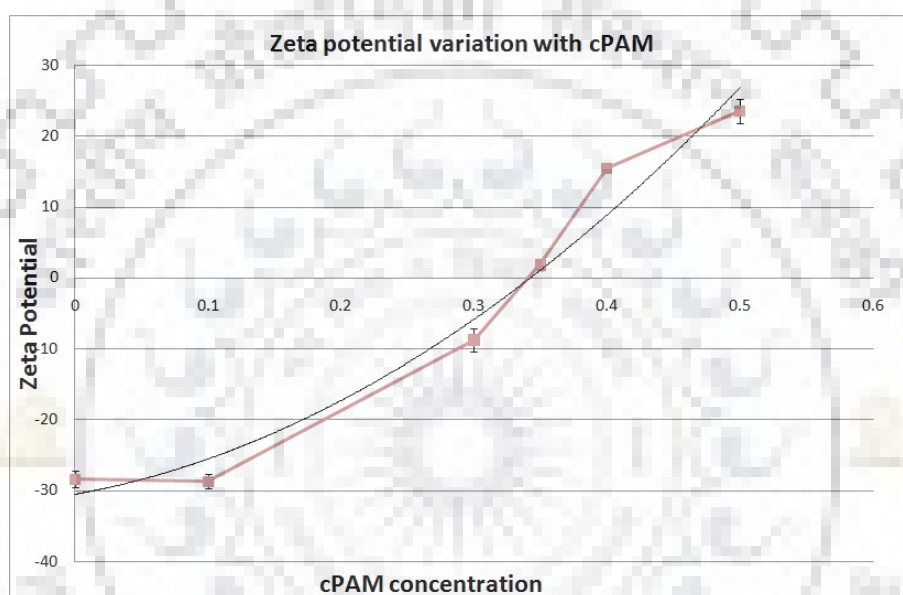


Figure 8: Variation in Zeta potential of FibCell (1 g/L) with cPAM

The fig.8 represents the change in zeta potential of FibCell wrt change in concentration of cPAM. The increment in zeta potential can be illustrated by a polynomial of second order. At a concentration of 0.35 % the zeta potential of FibCell exceeds zero. This values is taken as the middle value in order to observe a bigger picture about effects of cPAM on FibCell.

3.2.2 Cationic Starch

A medium D.S. pregelatinised cationic starch available in form of white powder was used during the conduct of experiment. Starch was supplied from Cargill star Bond.

The fig.9 indicates that there was a logarithmic increment in zeta potential of FibCell with variation

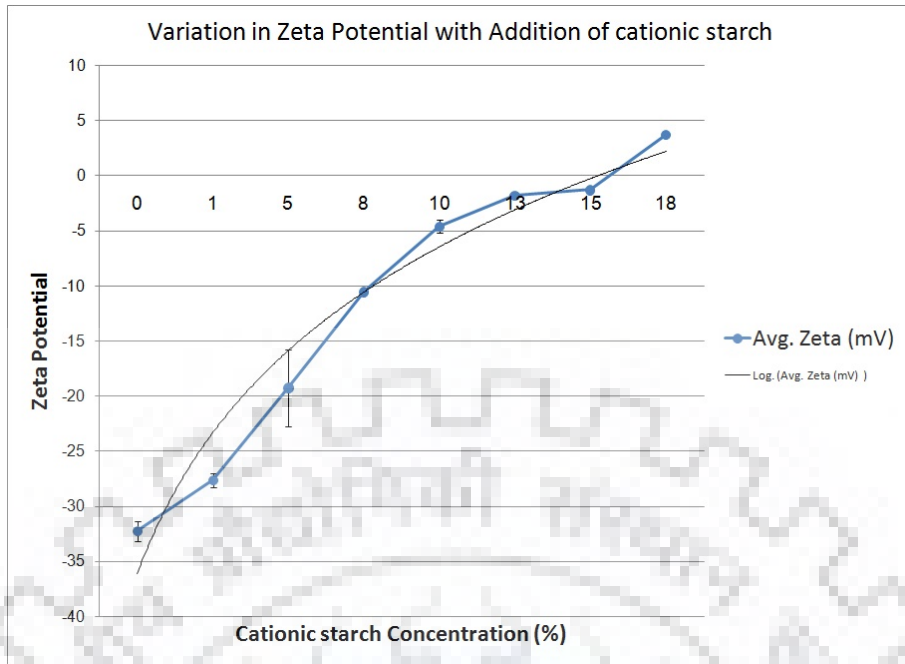


Figure 9: Variaton in zeta potential of FibCell (1 g/L) with cStarch

in cationic starch concentration. Initially there was a significant increase in zeta potential with addition of starch. But, as zeta potential tends toward zero the effect of starch also reduced. At about 15 % zeta potential of FibCell reaches -1.3 mV. It would not be economically feasible to use cationic starch more than 15 %. So, we considered 15 % as the upper limit for cationic starch.

Additive type	Min (%)	Mid (%)	Max(%)
CaCO ₃	0	5	10
cPAM	0	0.35	0.7
cStarch	0	7.5	15

Table 4: Limits of various additives used for analysis.

3.3 Design of experiments

Box-Behnken design was used in deciding the no. of trials required to be performed. So, that a robust model with relatively lesser no. of trials can be formed. A three level RSM design was considered. All design points, except the center points, are located at the center of the edges of the hypercube, and are also on the surface of a sphere. BBD is a good design for response surface methodology because it permits a good estimation of parameters of a quadratic model. When compared to other designing models it is found more efficient than other models. Number of trails required for this model are

$$N = 2k(k - 1) + C_o, \quad (1)$$

Where k represents the no. of factors and C_o represents no. of repetitions [20].

In our case we have three independent variables i.e concentration of three different additives and five response variables (zeta potential, dewatering time, etc). So, total no of experiments required to be performed are 15 in which 3 are repetitions or central points.

Repetitions are used to study the reproducibility of the results for same inputs (composition). It is important to perform them separately.

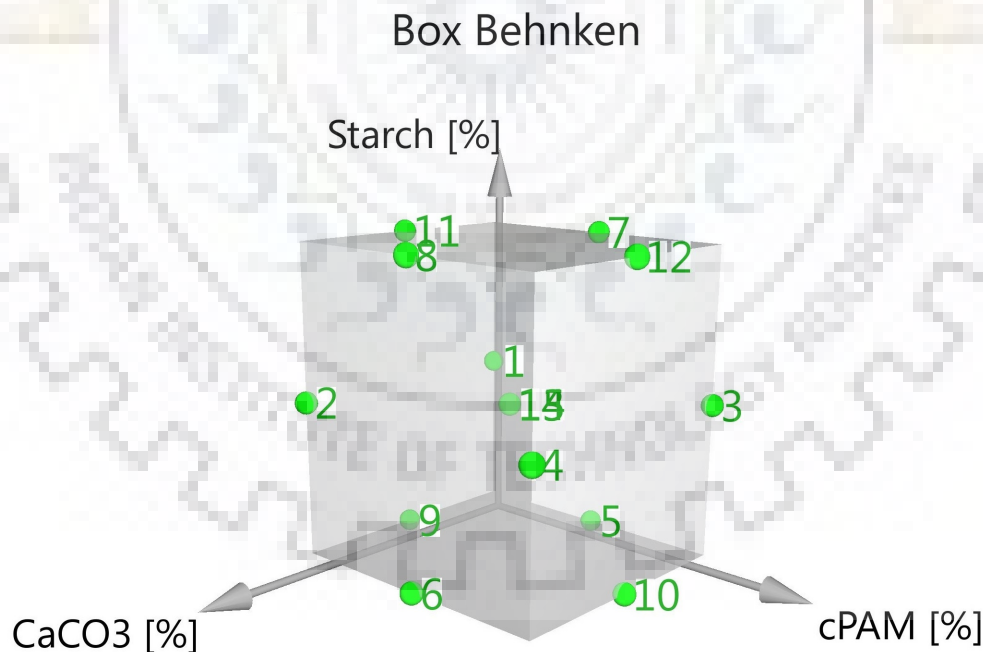


Figure 10: Garphical representation of Design region

Table 5 represents the fractionated factor plan to investigate the effect of constraints/input (concentration change) denoted by X_1, X_2, X_3 on the response variables/output (i.e. zeta potential, dewatering

time, tensile index, transparency, functional barrier and fat barrier). -1, 0, 1 represents lower, middle- and high level of inputs respectively.

Exp. No.	Constraints			Response
	CaCO ₃ (X ₁)	cPAM (X ₂)	Starch (X ₂)	Response variables (Y)
1	-1	-1	0	Y ₁
2	1	-1	0	Y ₂
3	-1	1	0	Y ₃
4	1	1	0	Y ₄
5	-1	0	-1	Y ₅
6	1	0	-1	Y ₆
7	-1	0	1	Y ₇
8	1	0	1	Y ₈
9	0	-1	-1	Y ₉
10	0	1	-1	Y ₁₀
11	0	-1	1	Y ₁₁
12	0	1	1	Y ₁₂
13	0	0	0	Y ₁₃
14	0	0	0	Y ₁₄
15	0	0	0	Y ₁₅

Table 5: Fractionated factor plan

3.4 Method of preparation

Highly fibrillated cellulose pulp, which was used for the study, had a dry fiber content of 4.25 g dry fiber/100 g. So, 705.88 g pulp (i.e. 30 g Dry fibre content) was taken and diluted with water to 2 L pulp-water suspension. This suspension was then disintegrated at 3000 rpm for 10 min. This disintegrated slurry was then diluted into 8 L of water so as to form a final suspension of consistency of 3 g/L.

3.5 Preparation of additives

Three additives are used namely CaCO_3 , Cationic polyacrylamide and Cationic starch as dewatering aids. All three additives were used in different concentration depending upon their effect on zeta potential of FibCell. Deionized water was used for diluting the additives

Preparation of CaCO_3

Carb-60, concentrated CaCO_3 solution with dry content 75 % was used. In order to form 1 % CaCO_3 solution 6.667 g Carb-60 was diluted in distilled water to form 500 g solution by weight.

Preparation of cPAM solution

As cPAM solution was highly viscous therefore, a low concentration is used while performing experiments. Firstly a solution with concentration 0.25 % was prepared which could be stored at low temperature for a period of 7-8 days according to guidelines provided by company. Then, while performing the trials a new solution with concentration of 0.02 % was prepared using distilled water.

Preparation of Cationic starch solution

Cationic starch was available in pure powder form. To form the solution first 250 g of distilled water was taken and stirred with magnetic stirrer at 300 rpm. Then, slowly a controlled amount of starch powder was released into the water in order to avoid any chunk formation. A total of 5 g of starch is used. When the complete starch is dissolved, resulting colloidal suspension is again diluted into distilled water upto a weight of 500 g which is subjected to continuous stirring at 200 rpm. So, a final starch solution with a concentration of 1 % is obtained.

3.6 Zeta Potential Measurement

MütekTM SZP-06 System Zeta Potential Analyzer of BTG Instrument GMBH was used for zeta potential measurement. The device is used to measure the surface charge in a fibrous suspension. In paper-making process it evaluates the performance of different wet strength agents by measurement before and after chemical contact. As it provides sample measurement without much sample preparation therefore it finds its place as a standard tool. In most of the paper industry it is successfully used to characterize the interaction of fillers, pigments and chemical additives with cellulose fibers. It can be used to determine optimum dispersing condition of additives. The device was designed for use at a temperature range of 10 °C to 40 °C (50 °F - 104 °F). The device can work at 230 V - 115 V at 50 Hz/60 Hz. Device should be placed on a stable and flat surface with a minimum of 8 inch distance from the walls.



Figure 11: MütekTM SZP-06 System Zeta Potential Analyzer

When mixed with water, cellulose fibers carry electric charge. At fiber surface, this electric charge leads to formation of a layer of oppositely charged ions so called counter-ions. A streaming current is measured in mV when these ions are separated from or sheared off the fiber. The fibers forms a plug remains at screen electrode and remain fixed while pulsating liquid flow shear off the counter-ions [21].

From the stock solution prepared earlier with a consistency of 3 g fibers/L, an amount approximately equal to $1/6^{th}$ of one kilograms is taken, so as to take 0.5 g dry weight only. This solution is then mixed with an appropriate quantity of additive (if any) and then stirred for 5 min duration at 150 rpm. After that desired quantity of another chemical additive is mixed and the resultant mixture is stirred at a constant speed of 150 rpm without giving any heat. This process is repeated n no. of times depending on the number of additives used so as to make a homogeneous mixture and give sufficient time for chemical contact. Now this mixture thus prepared is diluted with tap water to a weight of 500 g. This fiber mixture sample is placed under measuring cell made of plastic which is equipped with a ring electrode and a screen electrode. The sieve for our purpose was replaced by filter of grade 388. First of all instrument is calibrated using EL factor with the help of conductivity measurement through an external pre-calibrated conductivity meter.

$$EL\ factor_{new} = \frac{EL\ factor_{old} \times Conductivity\ Manual}{Conductivity\ Zeta} \quad (2)$$

After calibrating the instrument is then started. The vacuum extraction of the sample into the cell takes place where it forms a plug in the form of a thick cake of fibers. For forming a sufficiently thick and uniform cake a dwell time of 60 s is given before starting the measurement. An oscillating liquid flow is generated due to variation in pressure which is calibrated from -0.2 bar to -0.4 bar that induces a streaming potential. The output of device is printed using an integrated printer in device.

3.7 Water Retention Value

Water retention value was examined with the help of Dynamic Drainage analyzer as the time required to dewater Pulp from 1000 ml to 50 ml with consistency of 1g fibers per liter of solution.

The Dynamic Drainage Analyzer simulates conditions on paper machines and the shear can be precisely controlled to simulate pumps and other equipment. DDA has a turbidity sensor by which retention values are presented automatically. But, in our case turbidity sensor was not properly calibrated. Therefore, we measured by weight.

Drainage speed, initial drainage and temperature readings are presented for chosen time intervals. The effects of different pulp mixtures, degrees of refining, additions of chemicals and fillers on drainage conditions can be realistically studied.

The device used a supply pressure of 4.4 bar while the device was calibrated to generate a vacuum of 500 mbar for 600 s with a continuous stirring. A Pulp solution with consistency of 3 g fiber/L was

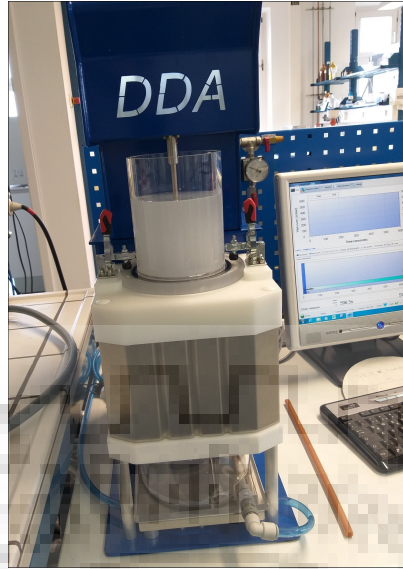


Figure 12: Dynamic Drainage Analyzer 5 - Pulp eye

prepared earlier. An amount approximately equal to $1/3^{rd}$ of a liter was taken out so as to have a solution with dry weight of 1 g. This stock is mixed with additive and subjected to a stirring of 150 rpm for a duration of 5 min respectively. So, that mixture thus formed is homogeneous with sufficient time provided for fiber-chemical interaction. As soon as stirring time completes the solution is diluted with tap water up to a weight of 1000g. This diluted mixture is stirred manually and directly transferred to DDA device. The device used a sieve of mesh width $0.025 \mu m$. As the device is started it lowers the piston and applies vacuum. The output is obtained in form of excel file. After dewatering a fiber cake was formed on the sieve. This fiber cake is further collected into a vessel using tap water. Then a vacuum of 8 mbar is used to separate fibers from water on filter paper of grade 388 whose dry weight is measured earlier. This separated cake on filter is dried in oven at a temperature of $105^{\circ}C$ for 15 min. The weight of sample is measured. Dry weight of fibers used in DDA is found, to spot any deviation in the quantity of fibers used or any fine lose.

3.8 Sheet Preparation

Sheets are formed in hand sheet former using a suspension with dry weight of 2.4 g/L. As we have previously prepared a stock solution of consistency 3 g/L.

$$x \times 3g/L = 1 \times 2.4g/L$$

$$x = \frac{3}{2.4} = 0.8L$$

So, 0.8 L of pulp from stock solution is taken by weight (as we have assumed that fiber solution has approximately the same density as of water). Precise amount of additive is added to the solution and the solution is stirred for 150 rpm for 5 min. This step is repeated for each and every additive used. The mixture is then diluted to a volume of 1 L (i.e. 1 kg). The mixture thus prepared is stirred manually to homogenize. This solution is poured into the hand sheet maker with a sieve having an mesh width of $25\ \mu\text{m}$.



Figure 13: Hand sheet forming machine

The sheet former works in seven steps which includes filling, pouring, aeration, still, drainage, vacuum and finally normal. The solution is poured only when there is at least 3 L of water and filled up to a maximum of 7 L. During all other steps a gap of 10 s is given. As the fiber size is too small it was not possible to remove the sheet from sieve directly without damaging it. Therefore, sheets are dried on dryer with sieve. The Dryer temperature was $94\ ^\circ\text{C}$ and sheet is dried for 10 min. Silicon protecting sheets were used instead of regular sheets because starch was bonding with the protecting sheet paper which makes it difficult to separate the two.

3.9 Properties

3.9.1 Grammage

Mass per unit area is defined as Grammage or basis weight of paper. Grammage is from french word used in English as it is for convenience purpose [22]. Grammage is also represented as GSM.

For determining the grammage of a sheet, a sheet of a is formed by sheet former is weighed by the well calibrated balance. The reading thus obtained is noted and searched against a standard table of weigh-grammage designed for sheets with area of 0.0317 m^2 . Thus grammage of sheet is obtained.



Figure 14: Balance

3.9.2 Thickness

Thickness of paper is measured as the perpendicular distance between two principal surfaces under prescribed condition. It is measured in mm or μm [23].

The samples to be tested are placed in preconditioned climate room ($23 \text{ }^\circ\text{C}$ and $50\% \text{ RH}$) for at least 48 hr. Micrometer uses a flat ground Pressure foot (circular movable face) and flat ground anvil (a circular fixed face) of size so that there is a continuous contact with pressure foot. In order to avoid deflections greater than 0.0025 mm a load of 1.5 Kg is applied on top of the frame. Initially the two faces are at zero. Either Manual or Automatic can be used to move up the movable face and then the sample is placed in between followed by moving down the movable foot. The reading is displayed on the digital screen in mm .



Figure 15: Micrometer

3.9.3 Tensile Strength

Tensile properties were calculated by the method as prescribed in ISO 1924-3. Tensile strength is maximum tensile force per unit width that paper and board will withstand before breaking under the conditions defined in this standard test method.

$$\text{Tensile Strength} = \frac{\text{Load at break}}{\text{Initial sample width}} \text{ in kN/m} \quad (3)$$

Tensile strength of a test piece is affected by factors like fiber length, fiber strength and bonding. It is very important during converting operations and hence prevent web breaking during web fed printing or web fed converting operations [24].

Tensile index is tensile strength divided by the grammage. Tensile index gives a better comparison between samples with different grammage.

$$TI = \frac{\text{Tensile strength}}{\text{Grammage}} \text{ in Nm/g} \quad (4)$$

Tensile energy absorption is amount of work done when a specimen stressed upto the failure under specific conditions under tension. It is measured in Joule/m^2 .

$$TEA = \frac{\text{rupture energy}}{\text{initial strip width} \times \text{initial test length}} \text{ in Joule/m}^2 \quad (5)$$

Test method:

The preconditioned test samples are taken and are cut into 10 strips of width 15 mm and length up to 100 mm. The testing is done on horizontal tensile tester with Constant rate of elongation. Tensile

tester is consisting of two clamping jaws both with line of contact for gripping the sample. The line of contact is perpendicular to the direction of the applied load for controlling and adjusting the clamping pressure.

The prepared strips are kept in between the clamping jaws, the automatic sample detector detects the sample quickly and allows the jaws to clamp the sample. Once the force is applied, one jaw will be moving with an elongation rate of 10 mm/min.

The test should be completed within 5 to 30 s.

3.9.4 Functional Barrier

Functional barrier provide protection against unauthorized substance through a functional barrier. It provides with an estimate of effectiveness of packaging material against loss of aroma. The functional barrier properties of Packaging against contaminants such as mineral oil saturated hydrocarbons (MOSH $C_{16} - C_{24}$) can be effectively studied using n-Hexane as a simulant (n-Hexane molar weight= 86 g/mol, Melting point= -94 °C, Vapor pressure= 160 hPA). Hexane Vapor Transmission Rate (HVTR) is determined using gravimetric method. The method uses a metal container (evaporation chamber) and a sealable closure with screws as shown in fig.16. The sample to be tested is placed in a hole of specific diameter [25].

The evaporation chamber consisting of evaporation cell, piece of sponge, rubber sealing. Prior to the testing sample is visually inspected for any crease, fold or inhomogeneity. The sample is cut into circular shape having diameter equal to outer diameter of rubber sealing (i.e. 68 mm approx) using scissors. First of all, the empty weight of the evaporation chamber together with sample is measured (W_0). Then, using one way pipette approximately 2 ml of n-Hexane is added to the sponge and sample is placed on the closure between the rubber sealing. The chamber is then closed by tightening the screw properly. So that the seal could be air tight. The weight (w) is measure using balance immediately after sealing the sample and recording time is recorded. Next weight measurement are taken after 2, 4, 6, 8 and 24 hr respectively. For safety purposed the experiment is always performed in fume chamber to allow gases to escape.



Figure 16: Evaporation Chamber, the device for the BSAF- HVPT

3.9.5 Fat barrier

Fat barrier or grease barrier is an important for certain packaging materials which is used for packaging of oily product. The main intention of this test is to estimate the level of grease or oily resistance provided by FibCell. It gives an estimate on how fast and how much simulated fat material can penetrate through the sheet.

According to DIN 53116 oil of palm kernel (Palmkernfett) can be used for the determination of grease-proofness of packaging material. The test instrumentation involves a heavy standard weight of about 523.77 g with glass plate at its base. Below the test sheet a clean paper view sheet is placed. On the sample sheet fat simulant is spread in a small rectangular area than a protector transparent film is placed on the fat layer and above that the weight is placed. The whole setup is placed on a horizontal table with a protected paper on it. After 24 hr time period the sample is checked for any stain on the view sheet. Grease-proofness of the Sample can be estimated by the size of stain on the view sheet.

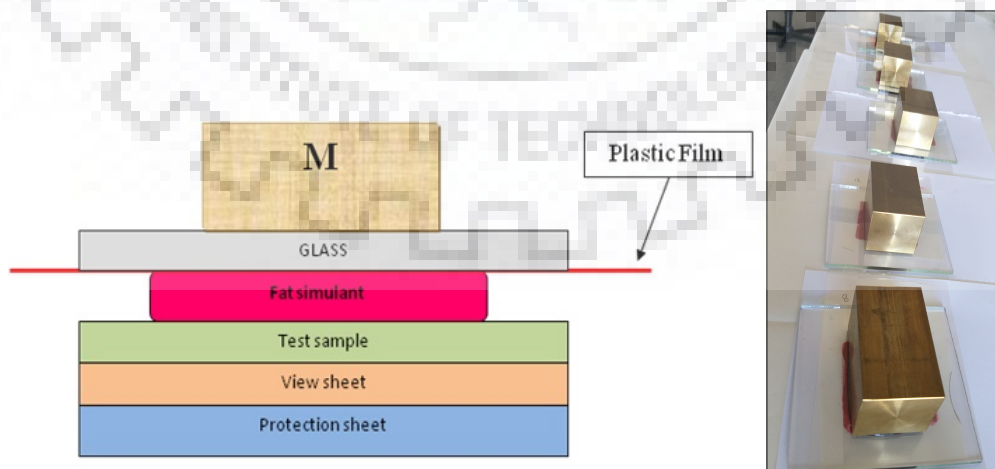


Figure 17: DIN 53116- Fat penetration testing

3.9.6 Optical Properties

Packaging function is not only limited to safe and sound delivery of product to the ultimate customer but also make the product look aesthetic and authentic. So, optical properties are of a great concern while designing packaging for a product. The good packaging material should have good optical properties so that it can be printed with desired design so as to make the product more attractive and cause impulse buying. For measuring optical properties spectrometer from L & W Elrepho was used. It complies with ISO 2469 and measures whiteness and brightness at D65 and C-illuminant. The device uses pulsed xenon lamp, D 65 - filtered. Firstly the device is calibrated using three standard calibration accessories which consist of two tiles (white and fluorescent reference standard) and a black trap. Five points of one experiment are tested so as to compensate for two-sidedness and in-homogeneity. D65/10° (illuminant/observer) was used for measurement of all parameters.



Figure 18: L&W Elrepho spectrometer

4 RESULT AND DISCUSSION

4.1 Model

A quadratic model was formed on Modde 12.1 using the data obtained by experiments in order to interpret the results. The software forms a quadratic equation for each responses selected and determine its dependence on different factors used.

4.1.1 Design Coefficients

Response	Significant model terms
Zeta Potential	CaC, cPam, Sta, CaC*CaC, Sta*Sta, CaC*Sta, cPam*Sta
Dewatering time	CaC, cPam, Sta, CaC*CaC, cPam*cPam, Sta*Sta, cPam*Sta
Tensile Index	CaC, cPam, Sta, CaC*CaC, cPam*cPam, Sta*Sta, cPam*sta
Transparency	CaC, cPam, Sta, CaC*Sta, cPam*Sta
Mineral Oil Barrier	CaC, cPam, Sta, cPam*cPam, Sta*Sta, CaC*Sta

Table 6: Significant Model coefficient terms

Response	Insignificant model terms
Zeta Potential	cPam*cPam, CaC*cPam
Dewatering time	CaC*cPam, CaC*Sta
Tensile index	CaC*cPam, CaC*Sta,
Transparency	CaC*CaC, cPam*cPam, Sta*Sta, CaC*cPam
Mineral Oil Barrier	CaC*CaC, CaC*cPam, cPam*Sta

Table 7: Insignificant model coefficient terms

Table 6 represents significant terms which have an effect (either positive or negative) on the respective response. These terms are included while drafting model for variable. Table 7 represents insignificant terms which are excluded while designing model for a response. Because these coefficient don't have any influence on output.

4.1.2 Summary of fit

The fig.19 represents the summary of fit for Box-Behnken model design for various response terms (i.e. zeta potential, dewatering time, tensile index, mineral oil barrier).

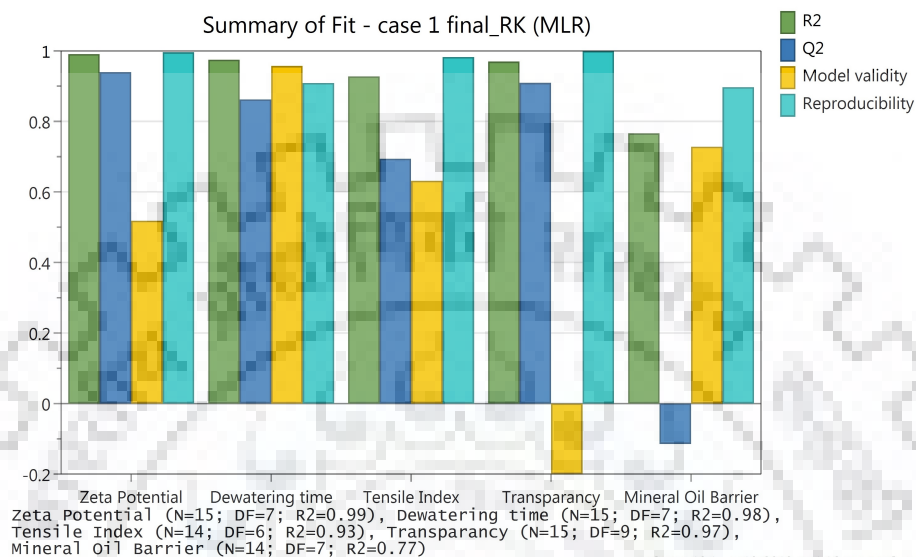


Figure 19: Summary of fit

The first bar in the Summary of fit plot is the R2, which is the fraction of the variation of the response explained by the model.

The second bar in the Summary of fit plot is Q2. The Q2 estimates the predictive ability of the model (also known as model predictive power), i.e., its ability to generalize to new, unseen data.

The third bar in the Summary of fit plot is the Model Validity. When the model validity column is larger than 0.25, the model has no lack-of-fit. This means that the model error is not significantly larger than the pure error.

The fourth bar in the Summary of fit plot is the Reproducibility, which is the variation of the response (often at the center points) under the same conditions as the total variation of the response.

It can be observed that for most responses all of the four bars are in good range except for mineral oil barrier (for Q2). Which can be explained as an effect as a result of the exactly same results obtained on repeated experiments. So, in general our model design is good for optimization and interpretation of our results.

4.2 Variation with Composition

According to the design of experiments trials were performed and different properties were measured. Each of the three additives selected has a unique effect in terms of altering these properties. Flocs were formed as a result of neutralization and patch formation. So, in order to understand the effect of additives, these variation results were studied.

4.2.1 Zeta Potential

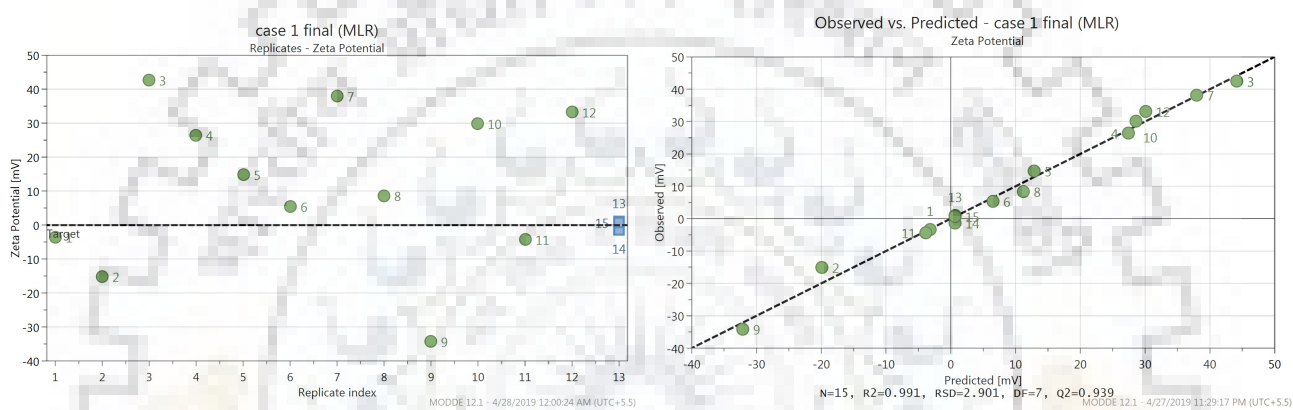


Figure 20: (a) Zeta potential Results , (b)Observed v/s Predicted Zeta potential value

The above fig.20a shows the results obtained by zeta potential experiments performed at random order according to the model. By considering fig.20b between observed vs predicted values it can be said that model is reliable.

From the Fig.21 following conclusions can be drawn.

As the amount of starch increase the value of zeta potential increase and same happens in the case of cPAM as can be seen when CaCO_3 is 0 %. But the increase in Zeta potential for cPAM is considerably higher than starch. This must be probably due to relatively higher charge density of cPAM than starch. When we consider the variation in CaCO_3 , with increase in Calcium Carbonate higher amount of other additives are required in order to reach the same value of zeta potential as in the previous condition i.e. 0 % CaCO_3 . And this trend continues itself as quantity of CaCO_3 is increased further. This is due to the blocking of possible adsorption sites by CaCO_3 . Target value is considered as 0 mV.

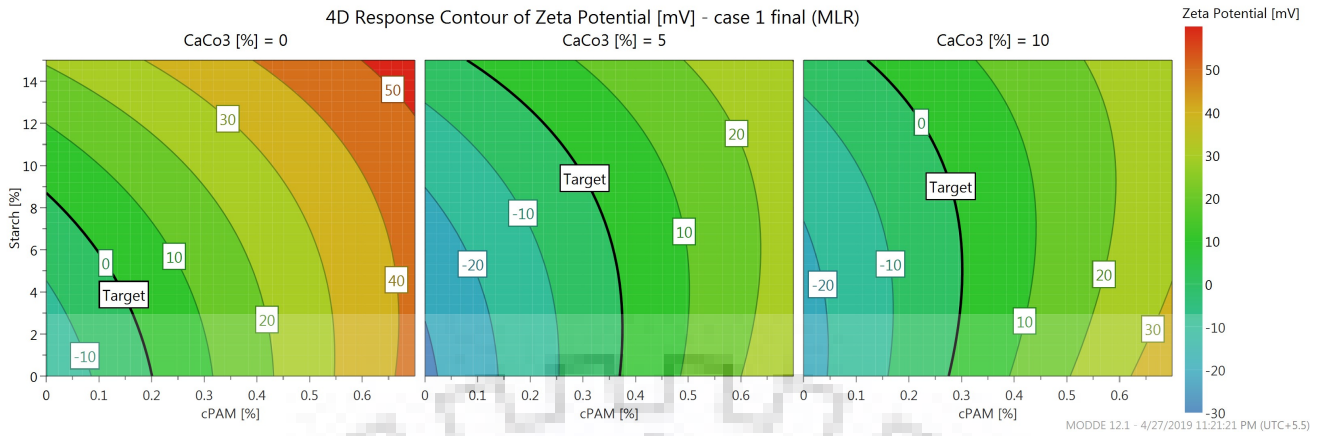


Figure 21: Variation in Zeta potential with change in cPAM and cStarch with constant CaCO₃

The below Fig.22 shows the predicted factor effect of a factor while all the other factors are kept constant to their average value. It can be clearly observed that as the slope of the curve is decreasing negative, hence as quantity of CaCO₃ increases, zeta potential decreases and tends towards zero, then it starts increase after 7 % concentration. In case of cPAM the slope is constant & positive and hence it shows a large linear increase in the Zeta Potential with 0 mV at around 0.35 % concentration. While

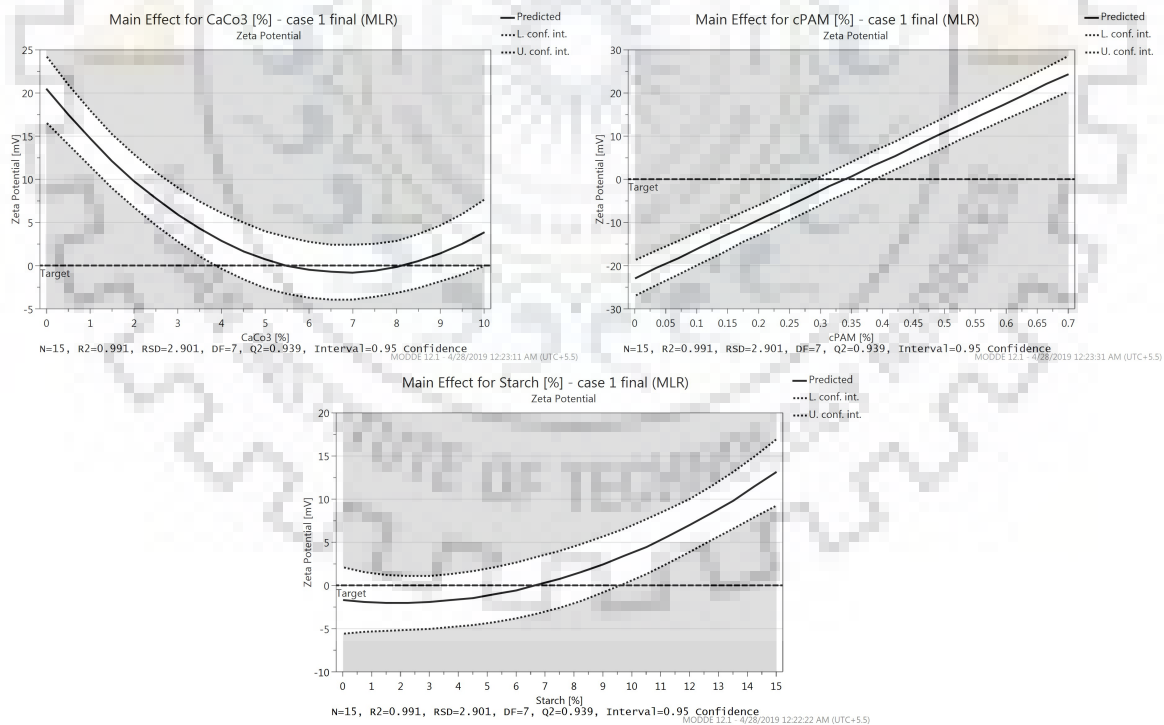


Figure 22: Predicted Main Factor Effect Curves (a) CaCO₃, (b) cPAM, (c) Starch for zeta potential

when factor effect curve of starch is observed then it show a non-linear (positive and increasing) slope and gives 0 mV zeta potential at 7 % concentration all the other additives kept constant at an average

value. The slow initial growth can be explained as a result of repulsion between similarly charged cPAM molecules.

4.2.2 Dewatering time

As a result of flocs formation dewatering time can be improved. The below shows the experimental values obtained for dewatering time. Point 13, 14 and 15 represents experiments with same composition performed at different time so as to study the reproducibility of results.

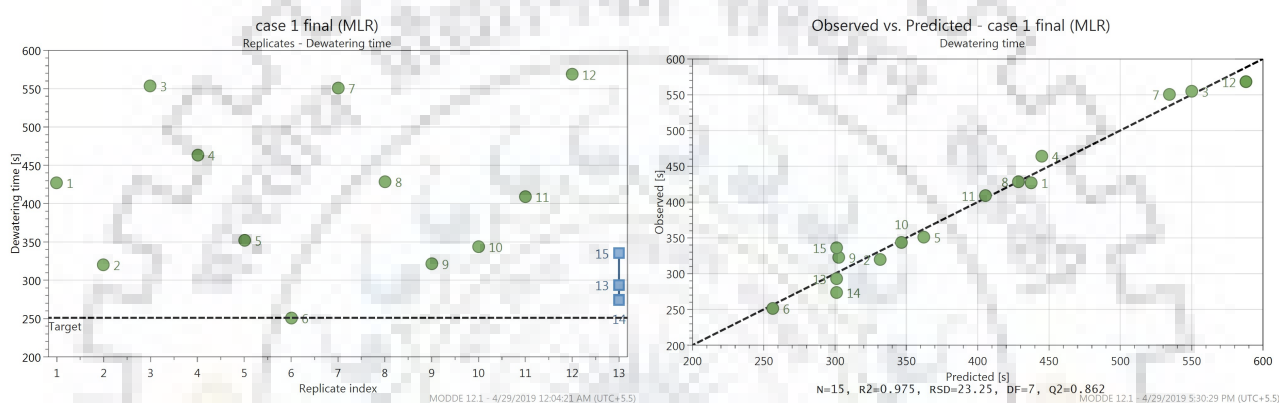


Figure 23: (a) Dewatering Time Values, (b) Observed vs Predicted Dewatering time

The fig.23b shows the curve observed vs predicted values model. Careful observation of this curve shows that experimentally observed values is very close to the value predicted by the model so the model is reliable.

The fig.24 show the variation of dewatering time with respect to change in CaCO_3 and cPAM while Starch concentration is kept constant (0, 7.5 and 15 %). Target is taken as 250 s. Following conclusions can be drawn.

From the fig.24a the desired region can be seen by blue ellipse bounded by target (time=250 s). As the concentration of CaCO_3 increases dewatering time decreases up to a minimum value at 7 % concentration, any further increase in concentration causes increase in dewatering time. Similar type of trend can be observed with variation in cPAM concentration. With increase in cPAM concentration dewatering time decrease up to a minimum value at 0.35 % concentration any further increase in concentration causes adverse effect on dewatering. This increase in dewatering time can be explained due to zeta potential reversal and viscosity change. In general when additives are added to fiber suspension viscosity increases. cPAM is an additive of very high viscosity. So, a concentration above 0.25 % will make the

solution thick enough to slow the dewatering process. As the concentration of starch is increased from 0 to 7.5 % it can be observed that there is no target region and dewatering time below 300 s cannot be achieved. Similarly after increasing the concentration of starch further to a concentration of 15 % it is not possible even to achieving the dewatering time of 300 s. As starch is available in clumps of molecules with relatively large size, hence it occupies most of the adsorption sites and reduce further flocs formation. So, this can be drawn that starch is showing adverse effect on dewatering time.

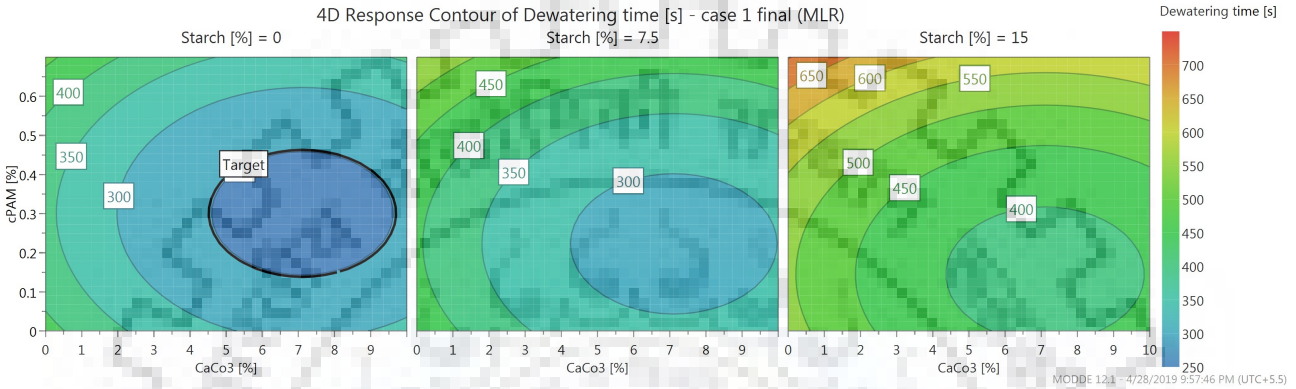


Figure 24: Variation of Dewatering time with change in CaCO₃ and cPAM at constant starch concentration

The fig.25(a), (b), (c) shows the factor effect CaCO₃, cPAM and Starch respectively while other two factors are kept constant at their average value. Following is observations can be drawn. Dewatering times shows non linear relation with all of the three additives. In the case of CaCO₃ the slope is negative and decreasing which reaches to zero at 7 % concentration, showing a minimum at this concentration and further increase will cause increase in dewatering time. While taking cPAM into consideration there is decrease in dewatering time evident by a decreasing negative slop curve which turns positive after 2.5 %. When observing the factor effect curve for Starch it is clear that starch does not help in dewatering at all. The slope is increasing and positive. Therefore, according to this curve any amount of starch added will reduce dewatering only.

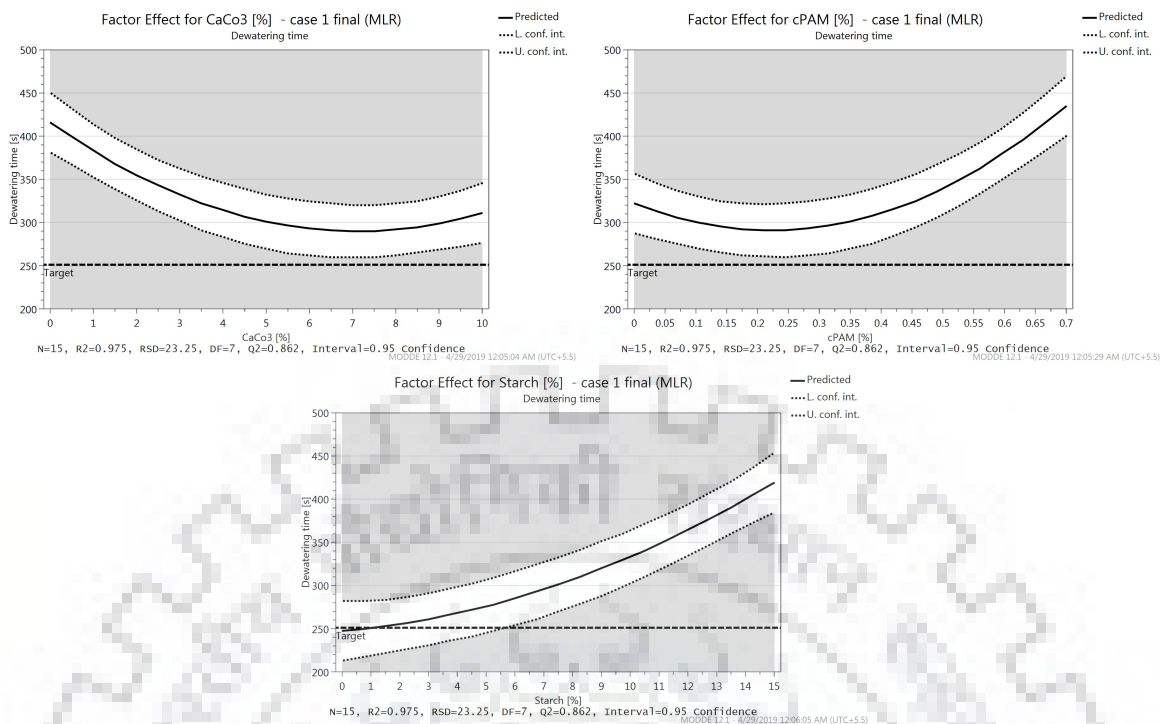


Figure 25: Predicted main effect curves (a) CaCO₃, (b) cPAM, (c) Starch for Dewatering time

4.2.3 Tensile Index

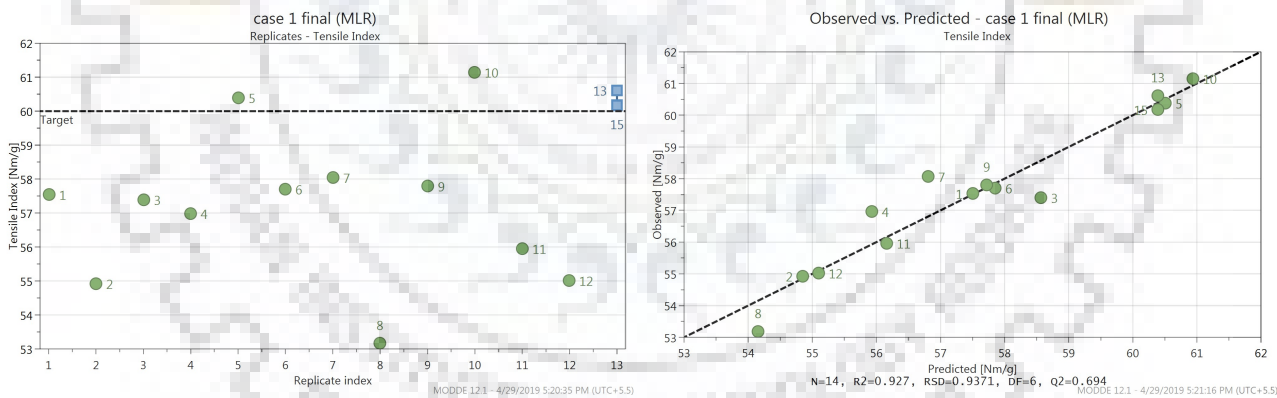


Figure 26: (a) Tensile Index values, (b) Observed vs Predicted values

The Fig.26(a),(b) shows experimental values obtained by tensile testing of the sheets produced for all trials performed (except trail 14) and observed vs predicted tensile index values respectively. And it can be concluded that model is fairly reliable.

Fig.27 represents change in tensile index with change in concentration of starch and cPAM while CaCO₃ is kept constant (0, 5 and 10 %). The target is represented by black curve line where tensile index attains a value of 60 Nm/g. It can be observed from the Fig.27(a) that as concentration of cPAM increase tensile index of sheets increases. This is primarily due to the formation of a strong

network of fibers and fines as a result of new bonds formation due to the action of positively charged additives. While as we move on y-axis it can be clearly observed that value of tensile index firstly increases by a little extent and then starts decreasing slowly with enhancing the concentration of starch. This is can be explained as the effect of difference in molecular size of the two additives. cPAM having relatively higher molecular weight undergoes adsorption at larger no. sites of fines causing the formation of a stronger network. While cationic starch causes a weaker network. With increase in additive concentration larger flocs (Macroflocculation) formation reduces the tensile strength. While as the concentration of CaCO_3 from 0 to 5 % clear increases in the strength can be observed same target value can be achieved with smaller concentration of cPAM. This can be explained due as the reduction in macro flocs formation due to the addition of CaCO_3 . But as the concentration is further increase than there is a noticeable detrimental effect on the tensile index. Even target value cannot be achieved as shown in fig.27.

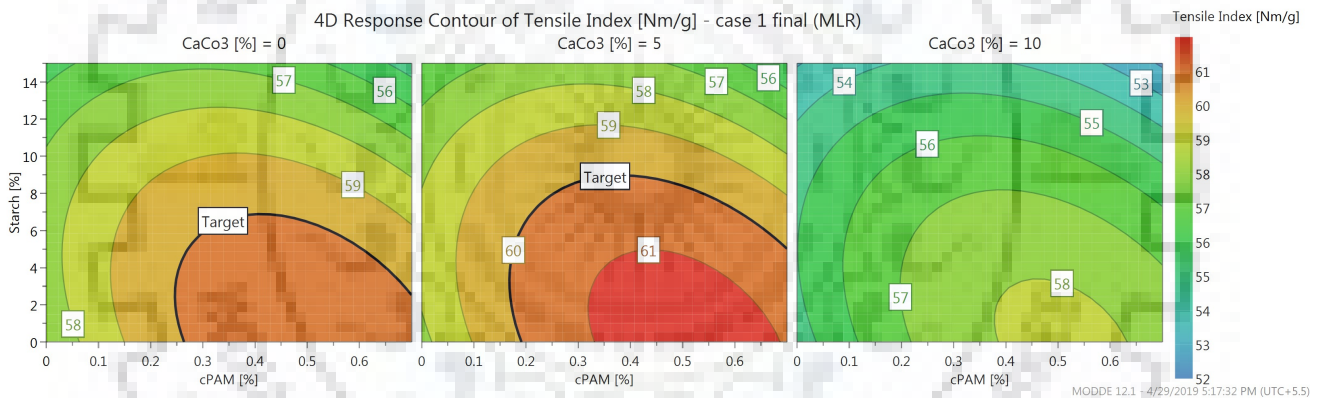


Figure 27: Variation in Tensile index with change in Starch and cPAM with constant CaCO_3

Fig.28 represents main factor effect on tensile index while other two additives are kept at their average value. When the concentration of CaCO_3 is increased there is slight increase in tensile index as the curve is having a very small positive and decreasing slope. And at a concentration of 4 % slope turns increasing and negative. So any further increase in CaCO_3 concentration will not be help in improving tensile index. The effect of cPAM shows a curve with a positives and decreasing slope with fairly high in terms of magnitude so initial quantity of cPAM will improve greatly till 4 % concentration. Any further addition of cPAM will reduce the strength as the slope changes negative and increasing. From the effect curve it can be seen that adding starch to the pulp can only decrease the tensile strength and no positive effect can be seen.

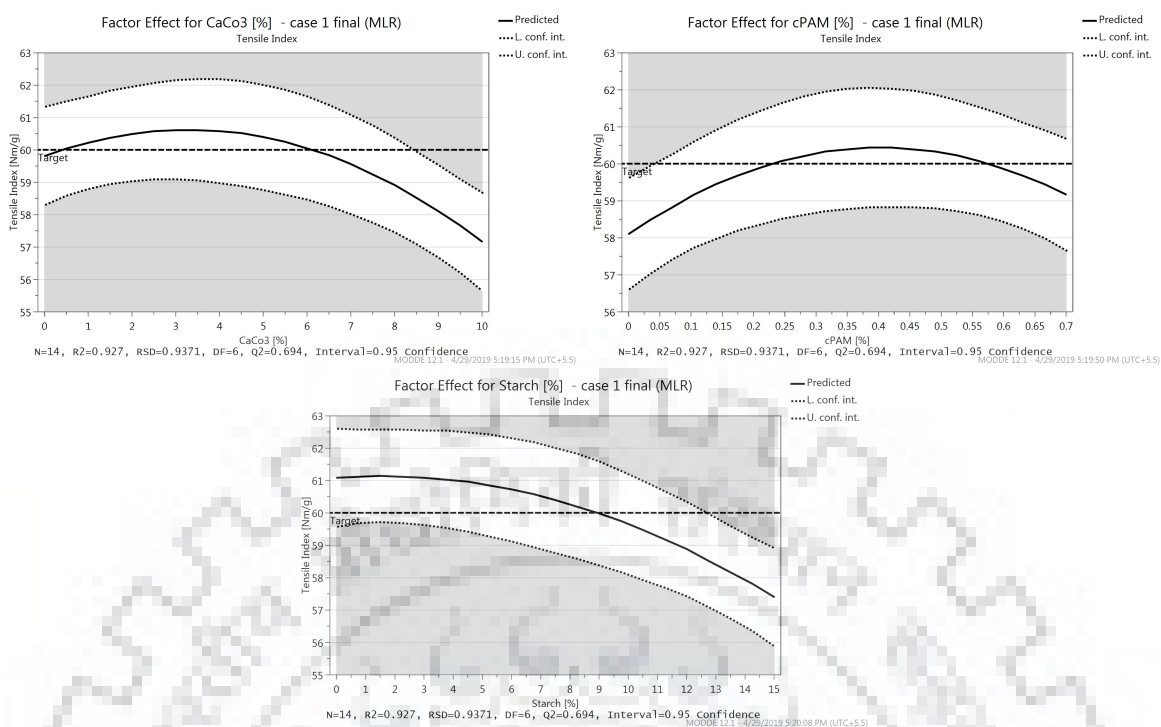


Figure 28: Predicted main effect curves (a) CaCO₃, (b) cPAM(c) Starch for tensile index

4.2.4 Transparency

Transparency is very important property while designing packaging material. For most of the packaging solutions ideal packaging material should be transparent enough to assist printing process except in case of see through packaging. If a high amount of macrofloculation is allowed then, sheet will appear to be a collection of cotton ball causing large variation in thickness and material.

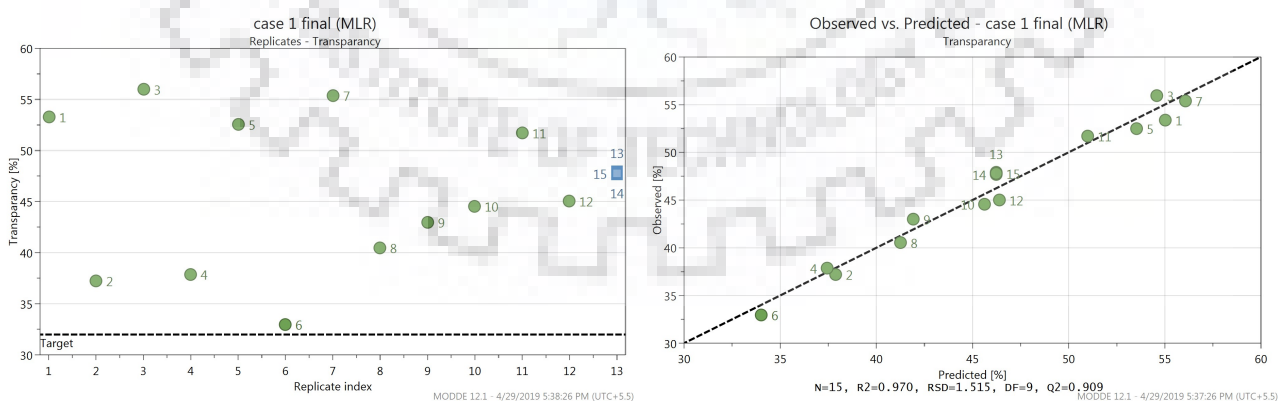


Figure 29: (a) Transparency values, (b) Observed vs Predicted Transparency values

When a wet end chemical additive is used to cause coagulation/floculation it effects the trans-

parency and cause cloudiness or lack in opacity. Fig.29a represents experimental transparency values obtained and Fig.29b represents observed vs predicted value curve so it can be assumed that model shows fairly reliable values.

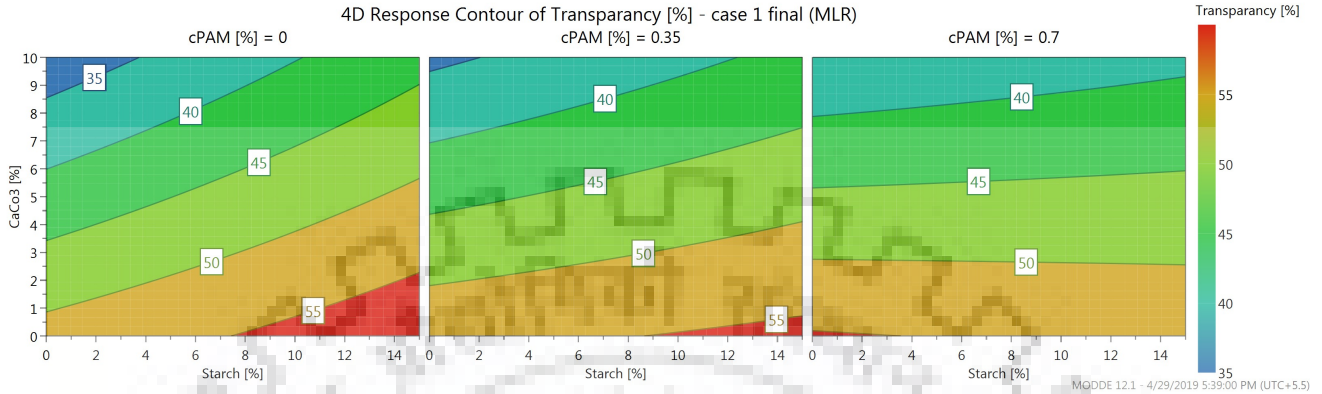


Figure 30: Variation in transparency with change in CaCO_3 and starch concentration at constant cPAM concentration

It was obvious assumption that CaCO_3 is going to decrease transparency of the samples formed and it is evident from the Fig.30a at 0 % cPAM and 0 % Starch i.e. on Y-axis as the concentration of CaCO_3 increases the value of transparency decrease up to a minimum of around 35 % at maximum concentration of CaCO_3 . Similarly, as we focus on X-axis it is evident that with increase in starch concentration increases the transparency with a maximum value of approx 55 % at 15 % starch. As the concentration of cPAM increased from 0 to 0.35 %. It can be observed that slope of lines on the concentration- transparency curve decrease with a very small magnitude and trend continues as the concentration of cPAM is further increased to 0.7 %. So, it can be concluded that cPAM concentration is having very weak incremental effect on the transparency. While the interaction between starch is having strong increasing effect on transparency. The increase in transparency can be explained as a result of flocs formation which cause more random distribution of fibers resulting in a cloudy sheet with transparent appearance.

Fig.31 represents the factor effect on transparency values while other factors (additives) are at a constant concentration at their average value. CaCO_3 effect curve shows a linear curve with negative slope with high magnitude which shows fair improvement in opacity of the sheet with CaCO_3 . The effect curve of cPAM shows that with other two at average concentration the interaction between the three causes the no significant effect of cPAM on transparency. While the effect curve for starch show a linear curve with positive slop relatively small as compared to CaCO_3 . Hence, any increase in starch

concentration will cause increase in transparency.

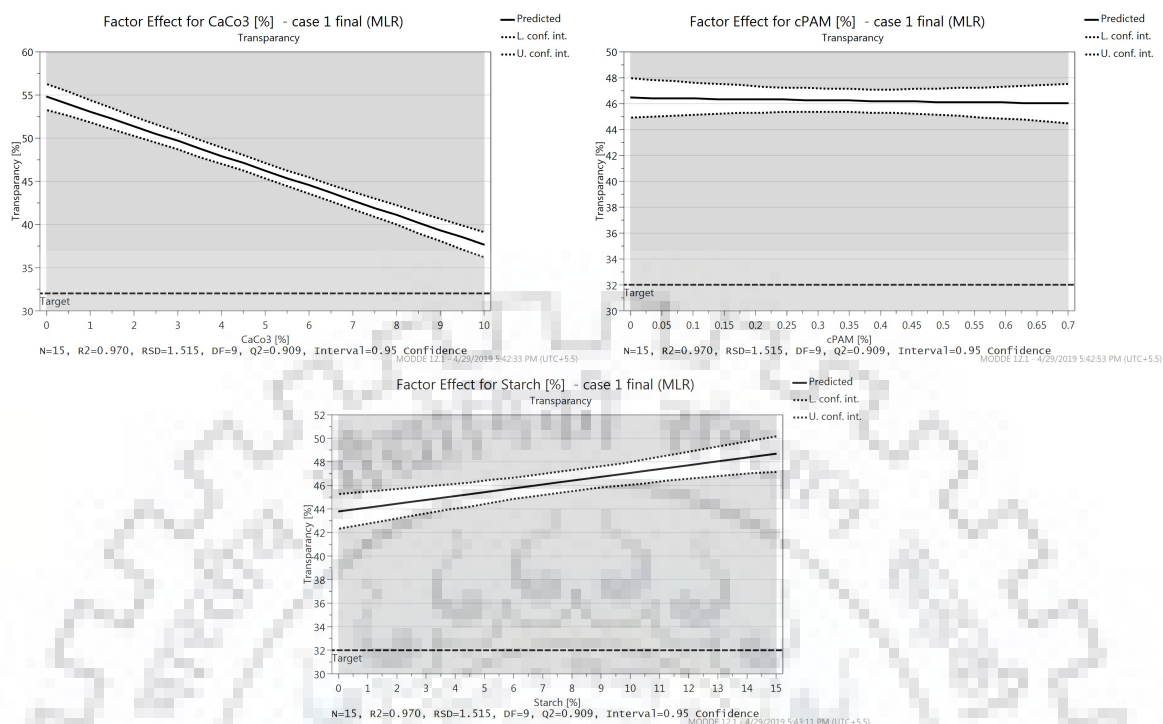


Figure 31: Predicted Main Factor Effect Curves

4.2.5 Functional Barrier

Barrier properties of Packaging against contaminants such as mineral oil saturated hydrocarbons is important for most of the food packaging because migration of such components cause unwanted effects on food as well as on human health. So, in this part of study the effects of different additives used in the study on the permeability of sheets produced are analyzed.

Below fig.32a shows the permeability values obtained by performing hexane vapor transmission test. The fig.32b represents relation between observed and predicted value through which it is clear that model produced is fairly reliable.

From Fig.33a following can be concluded.

As the concentration of CaCO₃ increases when cPAM and Starch concentration is 0 % i.e. on Y-axis, the value of permeability decrease from 20 to 15 g/m²d. On the other hand when we allow variation of starch keeping other two at 0% i.e. on X-axis, it can be clearly seen that the value of permeability decrease. On increasing the concentration of cPAM to 0.35 % the permeability is increased by a

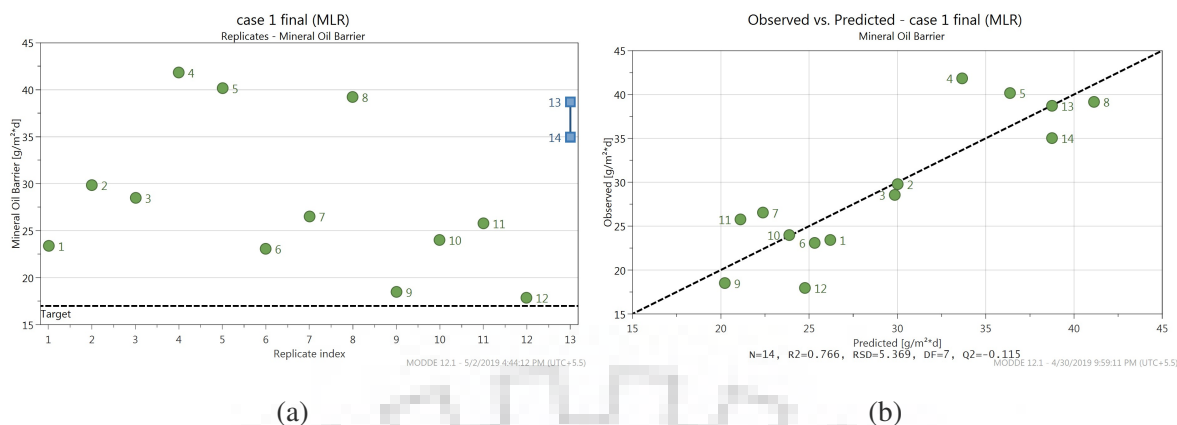


Figure 32: (a) Permeability values obtained, (b) Observed v/s Predicted HVMT values

quite large amount as seen in fig.33b same composition of CaCO₃ and Starch gives a higher value of permeability than in the previous case. Finally as the value of cPAM increased further to the highest level, 0.7 % then again it can be observed that there is a reduction in permeability as compared to the previous value. So, it can be concluded that there is a complex effect of interaction of different additives on permeability of FibCell sheets.

Fig.33(a), (b) and (c) represents surface curves representing as the ratio of additives decreases value of permeability increases. As the concentration of the two additives increases the quantity of n-hexane migrated increase. So, it can be said that interaction between additives is causing a negative impact on the gas permeability of FibCell. As the quantity of starch and cPAM increase permeability increases as a result of uneven distribution of material. When zeta potential crosses isoelectric point due to repulsion forces flocs start redispersing hence decreasing the permeability.

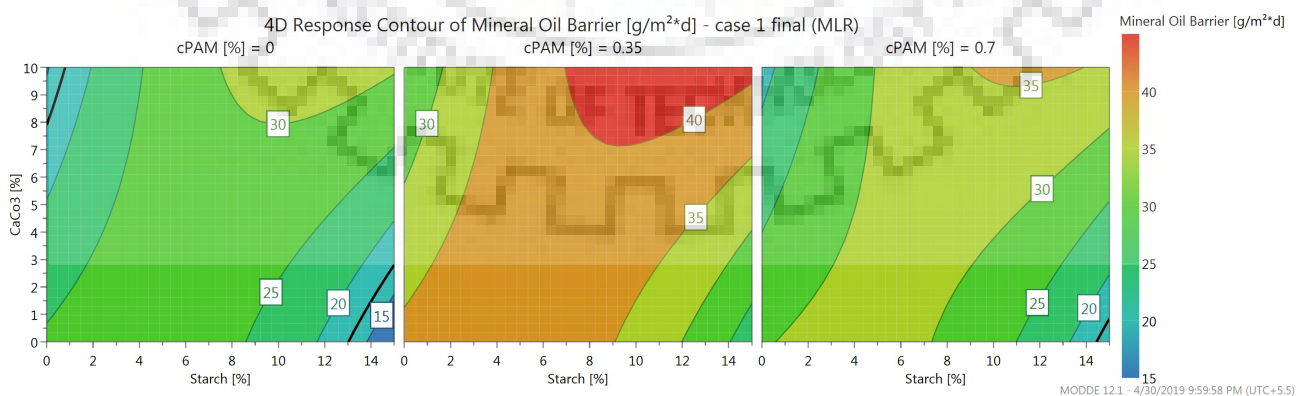


Figure 33: Variation in permeability with respect to change in concentration of CaCO₃ and Starch at constant cPAM

4.2.6 Fat Barrier

It is of great importance that packaging should be oil and grease resistant so that it can be used to store products which release fat as in the case of food products e.g. fish, Chips, Butter, Cakes, cookies. FibCell in its pure state i.e. without any additives used was very good in terms of fat barrier. So, in order to observe the effect of selected additives, different compositions (as our model design) were used and the resultant samples were subjected to fat penetration testing.

It was observed that there was no fat penetration found in any of the sample which shows that there was insignificant effect of additive composition on the fat permeability of the FibCell. Even though, it can be seen in fig.34 that in some of the cases fat spread all over the sample occupying a large area. So, it can be concluded that there was an increase in absorptivity of fat into samples. But still there was no fat transfer across the sheet even in these cases. Hence, it can be concluded that overall there was no major impact of using additives on fat barrier of FibCell.

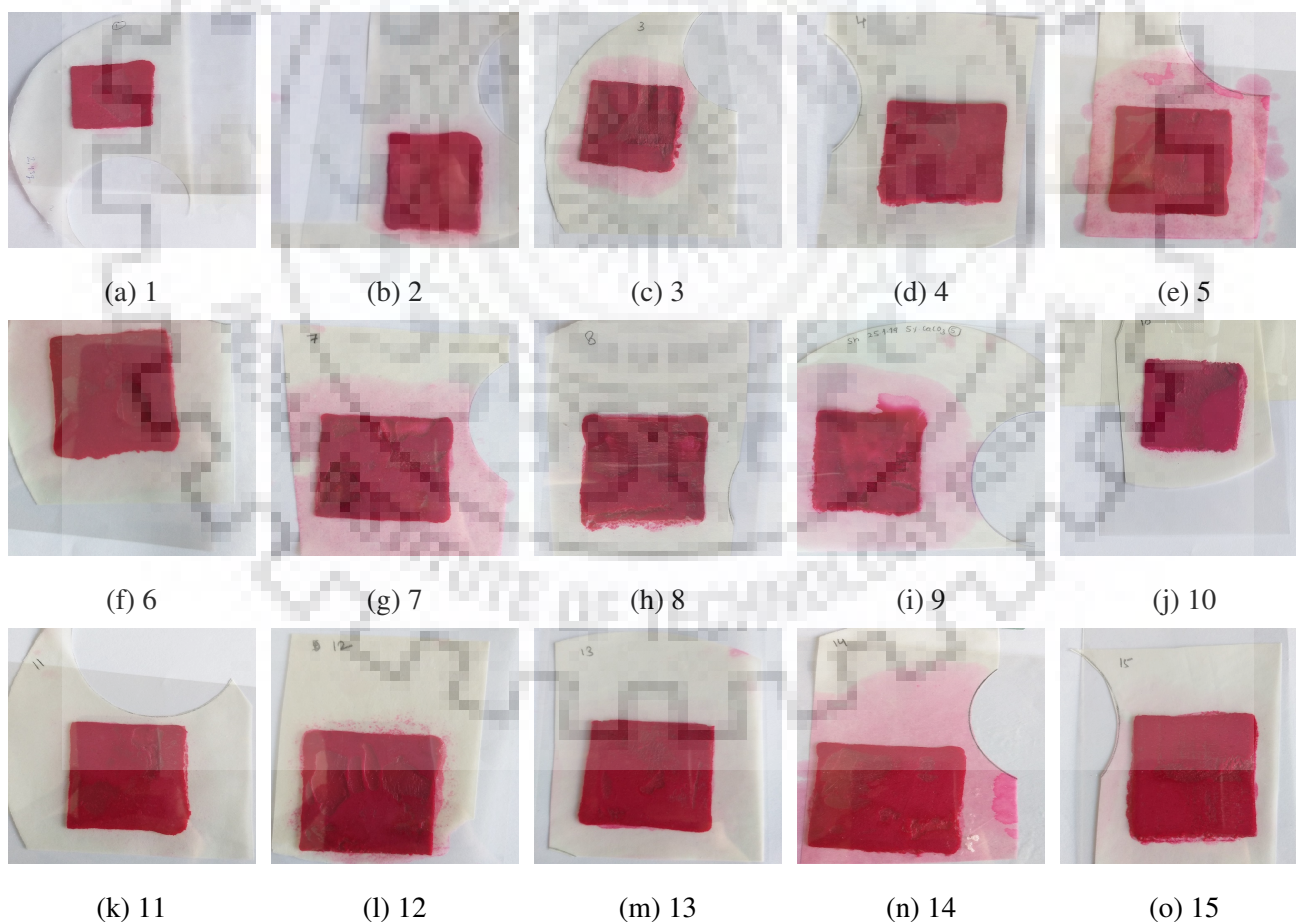


Figure 34: Results for Fat penetration test on FibCell sheet with different additive composition

4.3 Sources of Error

4.3.1 Variation in Conductivity

Fig.35a shows the conductivity of water supply measured during performing trials. It can be clearly seen that there was a significant variation in the quality of supplied water between trial. Sometimes there was variation in the quality of water during experimentation also.

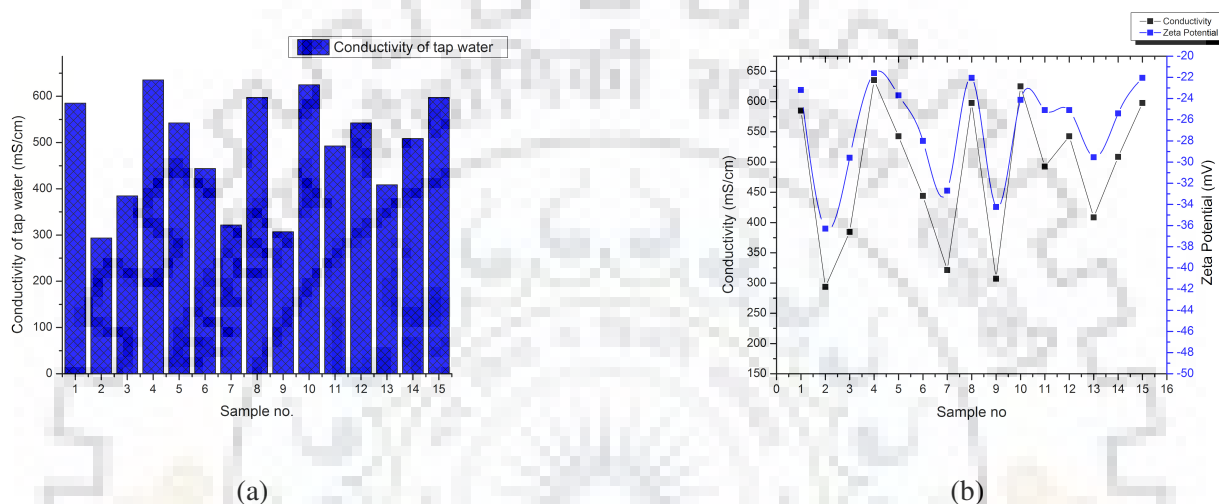


Figure 35: Variation in conductivity of water and FibCell solution with effect on Zeta Potential

Fig.35b show the relationship between conductivity of FibCell solution measured manually and Zeta potential of pure FibCell solution. And it can be clearly observed that there is a co-relation between two parameters. Zeta Potential increases with increase in conductivity. So, quality of water would be a possible culprit for fluctuation or error.

4.3.2 Two-sidedness

The sheets prepared are having two-sidedness i.e. one side was having more irregularities than the other which could cause deviation in optical properties, mineral oil barrier and Fat penetration properties as the side with higher irregularities is having more porosity than the later.

4.3.3 Clogging

During preparation of sheets the aperture size of mesh was found to be reduced due to the aggregation of starch particles on the mesh after performing 2-3 experiments. This causes delay in dewatering during sheet making. See fig.36, deposition of starch is evident from decreased transparency in the mesh. In order to remove the starch deposited on sieve it is placed in sonicator bath at 40 °C for 15 min.

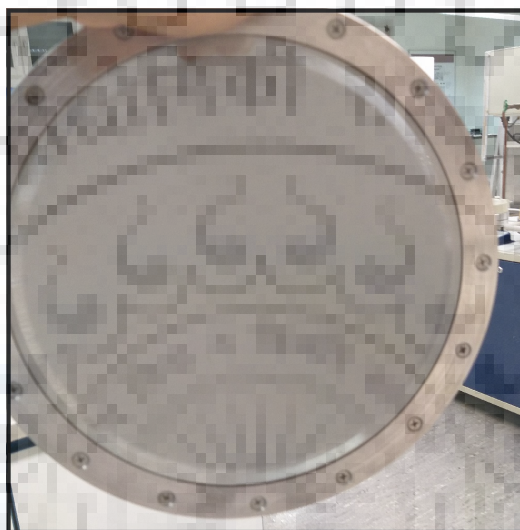


Figure 36: Opaque Mesh due to starch clogging

4.4 Optimization

The model thus designed for different objective functions (response) using Box-Behnken is optimized using optimizer tool in modde program.

4.4.1 Objective

Table 8 shows objective function used for optimizer. Zeta potential as its value was depends on water quality is excluded. Transparency is excluded from the optimized result as well because if it is possible to obtain a good functional barrier with high transparency then it would be fine. So, transparency is not the main concern response. Dewatering time and mineral oil barrier are aimed for minimum value. While tensile index is aimed for a maximum value. Pred. min and pred. max values shows the

maximum and minimum values that can be obtained using different combinations.

Response	Criterion	Min	Target	Max	Pred. min	Pred. max
Zeta Potential	Predicted				-32.2872	44.1313
Dewatering time	Minimize		0	400	234.206	589.513
Tensile Index	Maximize	53	60		51.9664	61.6851
Transparency	Predicted				32.3239	55.8012
Mineral Oil Barrier	Minimize		0	22	12.0699	38.74

Table 8: Objective for optimizer

4.4.2 Iterative solutions and sweet spot

Table 9 represents a list of optimized iterative solutions obtained using the modde program. It can be clearly seen the best run is solution no.8 with least probability of failure with all other conditions optimum i.e. minimum dewatering time (297.9 s) with a reasonable compromise in tensile index (55.7 Nm/g) and functional barrier properties (16.1 g/m²d).

Sr. No.	CaCO ₃	cPAM	Starch	Zeta Potential	Dewatering time	Tensile Index	Transparency	Mineral Oil Barrier	Iterations	log(D)	Prob. of failure
1	8.74	0.000214	1.91E-05	-28.07	298.3	55.7	34.6	16.1	583	-0.31	0.13
2	9.90	0.7	0.0005	36.40	354.9	57.8	36.1	18.5	430	-0.28	0.3
3	4.16	8.61E-05	15	-1.29	416.2	56.3	52.2	19.5	323	-0.15	0.28
4	1.33	7.76E-07	7.11E-05	-23.55	375.0	57.7	49.1	24.3	602	-0.13	0.15
5	8.63	0.000737	8.84E-05	-28.32	297.2	55.8	34.8	16.2	570	-0.31	0.13
6	9.86	0.7	0.000822	36.24	354.3	57.8	36.1	18.5	248	-0.28	0.3
7	8.73	8.02E-05	0.000106	-28.10	298.3	55.7	34.6	16.1	623	-0.31	0.13
8	8.69	1.46E-05	0.000738	-28.22	297.9	55.7	34.7	16.1	711	-0.31	0.12
9	8.69	5.91E-05	7.54E-05	-28.23	297.9	55.7	34.7	16.1	678	-0.31	0.13
10	9.84	0.697709	0.000152	35.99	352.9	57.9	36.2	18.6	487	-0.28	0.31
11	8.67	4.38E-06	0.00135	-28.28	297.8	55.7	34.8	16.1	531	-0.31	0.13
12	9.79	0.699821	2.17E-05	36.00	353.4	57.9	36.3	18.6	532	-0.28	0.3

Table 9: Iterative solutions of optimizing

Fig.37 is a graphical representation of iterative solution obtained using optimiser tool. The green region

represents the area where all the criteria or limits are satisfied. Dewatering time max represents the line below which dewatering time is 400 s and keep on increasing. similarly mineral oil barrier max and tensile index min represents limiting criteria for respective properties.

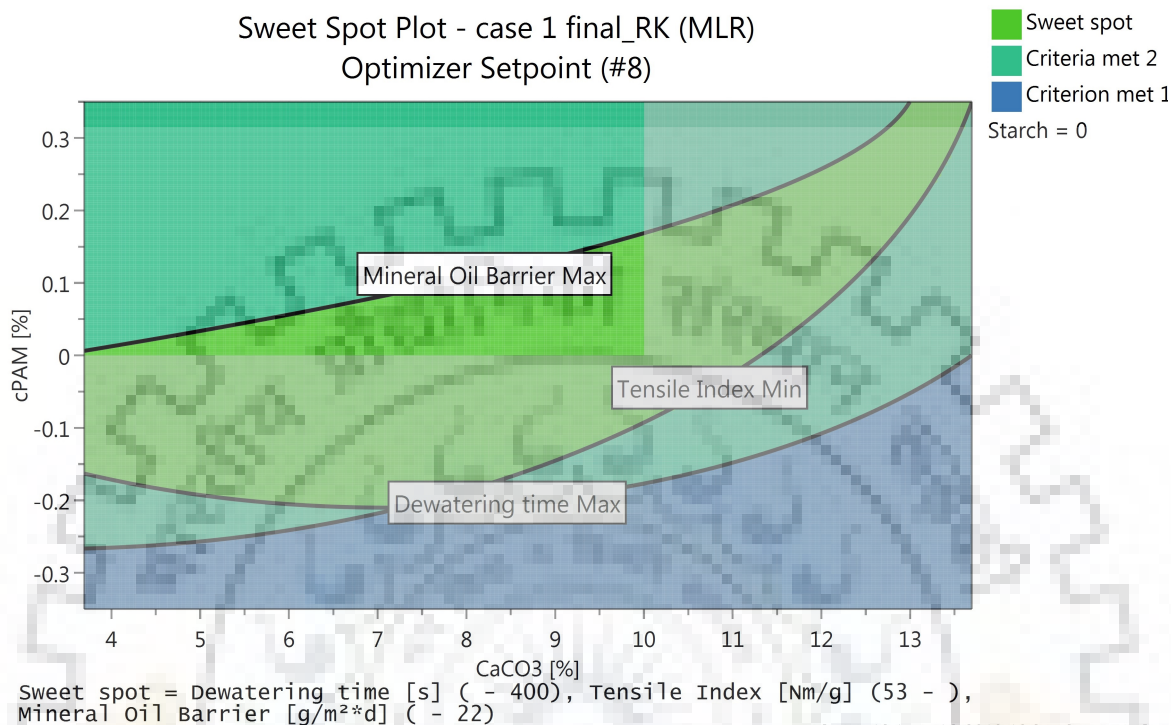


Figure 37: Graphical representation of iterative solution

Property	Pure FibCell	Iterative result	Experimental Value	prediction error	% Increment
Zeta potential (mV)	-32.25	-28.22	-22.5	-25.4	30.23
Dewatering time (s)	357	297.9	312	4.8	-12.61
Tensile index (Nm/g)	66.526	55.7	56.90	2.1	-14.47
Transparency (%)	62.50	34.7	36.30	4.4	-41.92
Mineral oil barrier (g/m ² d)	24	16.1	25	35.6	4.17

Table 10: Comparison between pure FibCell, Simulated result, Experimental result

From table 10 it can be observed that there was a reduction of 12.61 % in dewatering time while 4.17% increase in n-hexane transmission and 14.47% reduction in tensile strength which can be considered tolerable for packaging application.

4.5 Conclusion

The results of this study demonstrated that the zeta potential plays an important role in flocculation and dewatering behavior of FibCell as well as zeta potential is not an absolute factor and can be altered with water quality. FibCells were flocculated by addition of additives, and this impact on the network strength and dewatering ability of the suspension was investigated. Using a suitable combination of chemical additives, it is possible to substantially increase the drainage. At the optimum point there was a reduction of 12.61% reduction in dewatering time with a good 41.9% decrease in transparency and tolerable amount of reduction in permeability. From the study it can be concluded that it is possible to reduce dewatering time with a reasonable compromise between other properties and hence, there is a possible potential for FibCell to be used for Industrial purpose.



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