



8-2002

A study of nonwoven composites

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To the Graduate Council:

I am submitting herewith a thesis written by Shaker Gaddam entitled "A study of nonwoven composites." I have examined the final electronic copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in .

Dr. Larry C. Wadsworth, Major Professor

We have read this thesis and recommend its acceptance:

Dr. Randall R. Bresee, Dr. Gajanan S. Bhat

Accepted for the Council:

Carolyn R. Hodges

Vice Provost and Dean of the Graduate School

(Original signatures are on file with official student records.)

To the Graduate Council:

I am submitting here with a thesis written by SHAKER GADDAM entitled “A study of nonwoven composites”. I have examined the final electronic copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science with a major in Textiles, Retailing & Consumer Sciences.

Dr. Larry C. Wadsworth

Major Professor

I have read this thesis

And recommend its acceptance

Dr. Randall R. Bresee

Dr. Gajanan S. Bhat

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**Vice Provost and
Dean of Graduate Studies**

(Original signatures are on file with official student records.)

A STUDY OF NONWOVEN COMPOSITES

A Thesis

Presented For The

Master of Science

Degree

THE UNIVERSITY OF TENNESSEE, KNOXVILLE

Shaker Gaddam

August 2002

DEDICATION

I dedicate this thesis

**To Him
To Whom
The Glory Is Justly Due**

And

To my parents

Mr. G. Narsimha Reddy and Mrs. Vani

and

my brothers

Mr. G. Kishore and Mr. G. Rakesh

ACKNOWLEDGEMENTS

I would like to thank my advisor, Dr. Larry C. Wadsworth for his continued guidance and encouragement through out my studies at the University of Tennessee. Sincere appreciation is extended to Dr. Randy. R. Bresee and Dr. Gajanan Bhat for being on my committee and for their advice and guidance. I would like to thank Rongguo Zhao, Jack and other lab technicians at TANDEC for their helping hand in processing the polymers and producing the samples. My sincere appreciation to the statistical consultants Mike Newman and Mike O' Neil for their support in analyzing the results. I sincerely thank all my colleagues and friends at TANDEC for their timely help in many ways.

ABSTRACT

Preliminary work on nonwoven composites at The University of Tennessee showed that spunbond/meltblown (SM) and spunbond/meltblown/spunbond (SMS) nonwoven composites containing side-by-side (S/S) bicomponent PP/PE fiber meltblown webs had lower flexural rigidity than 100%PP SB webs and that the SM composites had discernibly softer hand than did the composites made from 100% PP in the meltblown component.

This study further optimizes the production and processing parameters of the SM and SMS laminates containing MB webs with different ratios of the bicomponent polymer pairs PP and PE. The resultant laminates were tested for barrier performance, tensile strength, hydrostatic head (HH), air permeability (AP) and flexural rigidity (FR) properties. Response surface modeling was used for the analysis of the HH, AP, FR and tenacity of both the SM and SMS laminates. The effect of % polypropylene in the bicomponent meltblown web on the properties of the laminates was investigated using qualitative and statistical analysis methods.

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LIST OF ABBREVIATIONS

1. SB	Spunbond
2. MB	Meltblown
3. PP	Polypropylene
4. PE	Polyethylene
5. SM	Spunbond/Meltblown
6. SMS	Spunbond/Meltblown/Spunbond
7. Bico	Bicomponent
8. INDA	Association of Non-woven Fabrics Industry
9. ASTM	American Society for Testing and Materials
10. HH	Hydrostatic Head
11. AP	Air Permeability
12. PLI	Pounds per Linear Inch
13. SEM	Scanning Electron Microscopy
14. SAS	Statistical Analysis Software
15. GLM	General Linear Model
16. RSM	Response Surface Methodology
17. DCD	Die-to-Collector Distance
18. LLDPE	Linear Low Density Polyethylene

CHAPTER I

INTRODUCTION

“According to the Nonwoven Fabrics Handbook, nonwoven composites refers to the products produced by a combination of one or more nonwoven fabric/technology with other materials/technology to make a better performing whole with the properties of the parts. The composites can be produced from the combination of any of the webs of spunbond, meltblown, wet-laid, dry-laid and other webs produced from nonwoven manufacturing processes” [5]. The laminate may contain two or three or more layers of nonwoven webs resulting in a laminated web, which combines the properties of the layers, used in the manufacture of the composite. Researchers have been studying laminates, according to Michael Jacobson, since the mid 1980’s [5]. During the past decade, nonwoven composites have drawn great attention from both industry and universities, as evidenced by the rapidly growing number of patents. The joining of different layers/fabrics/technologies has been a point of interest for many researchers and the present study combines the spunbond (SB), meltblown (MB) webs produced on the SB machine during the production of SB fabric. The SM and SMS laminates combine the filtration properties of the ultra fine MB fibers and the strength of the SB fibers. The MB webs were produced on the 24-inch Reicofil® bicomponent (bico) fiber MB machine in The Textiles and Nonwovens Development Center (TANDEC), located at The University of Tennessee. Bicomponent MB webs of various percentages of PP and PE were produced to study the effects of the proportion of PP and PE in the bico pairs on the

bonding performance and physical and barrier properties of the SM and SMS laminates. The various percentages studied were: 0%PE/100%PP, 25%PE/75%PP, 50%PE/50%PP, 75%PE/25%PP and 100%PP/0%PE. The MB webs and SB webs were laminated on the Reifenhäuser SB machine at different conditions of temperature and pressure, with the temperatures of 250, 275 and 300° F and with the calender nip pressures of 240, 270 and 300 PLI. The SB fabric take-up speed was maintained at 60.3 m/min for all trials. To understand and investigate the effect of temperature, pressure, percentage of PP in the bico MB web and basis weight of bicomponent on the performance of the laminated webs, the following properties of the laminates were determined: basis weight, air permeability, thickness, flexural rigidity, hydrostatic head and tensile strength. Response Surface Methodology (RSM) was utilized to analyze the performance properties and to optimize the processing parameters.

CHAPTER II

LITERATURE REVIEW

Spunbond Technology:

Spunbond (SB) and meltblown (MB) technologies are two of the most rapidly growing nonwoven technologies. The SB process involves the extrusion of continuous filaments from a spinneret. The extruded filaments are kept separated until solidified by air quenching. After the quenching stage, the SB filaments are rapidly drawn by air drag friction or by mechanical drafting rollers. The filaments are then deposited in a random orientation onto the collecting belt. Thermal calendaring through air bonding, needle punching and other mechanical or chemical means then bonds the filament webs. SB filaments generally have large average diameter (e.g. 12-50 microns, typically 15-35 microns) that are heavier and stiffer than MB fibers (e.g. 0.5 to 10 microns, typically 2-4 microns). Spunbond fabrics generally have the following properties:

- A) Random fibrous structure and in general the web is white with high opacity.
- B) Typical basis weight ranges are 10 – 200 g/m²
- C) Fiber diameter ranges between 15 and 35 μm
- D) Web thickness range between 0.1 and 4.0 mm, typically 0.2-1.5 mm.
- E) High strength to weight ratio.
- F) High liquid retention capacity due to high void content.

Melt blowing technology:

Melt blowing is a process for manufacture of nonwoven fabric in which thermoplastic polymer is extruded from a die tip having a row of spinneret orifices with typically 25-35 holes/inch. The fibers exiting from the die tip are contacted with converging sheets or jets of hot air to stretch or draw the fibers down to ultra-fine diameters typically ranging between 2-4 μm . The fibers are then deposited onto a collector in a random manner and to form a nonwoven fabric. The fibers in the web are usually self-entangled enough that additional bonding is not required. The melt blowing process consists of the following elements: extruder, metering pumps, die assembly, compressor or blower, air furnace and air delivery system to die.

Bicomponent fibers:

Bicomponent fibers (bico) are composed of two or more polymers of different chemical and/or physical properties extruded from the same spinneret orifice with both polymers within the same filament [18]. The first commercial bico was DuPont's "Cantrece", which was not commercially successful. Later ICI or British Nylon Spinners developed a bicomponent fiber named "Cambrelle", a thermally bonded Nylon 66/Nylon 6 bico, which is used in shoe interlining applications. Now the United States produces 60 million pounds of bico fibers annually with Hoechst Celanese being in the lead. The other U.S producers are BASF Corporation, DuPont Company, Fiber Innovation Technology, Intercontinental Polymers, KoSa, and Solutia. Bico fibers can be produced as very fine fibers with any cross sectional geometry, and they can be thermally bonded. The difference in melting temperatures

of the two component polymers is made use in forming self-bulking or self-crimping fibers. The properties of polymers can be best exploited by using bicomponent fiber technology. The general cross sections and geometrical shapes of bicomponent fibers produced are described below:

1. Side-by-side fibers:

The components used in the bicomponent fiber production should have good adhesion to each other unless it is desirable to subsequently subject the fibers to mechanical action such as hydro entanglement or chemical treatment such as using solvents to cause the fibers to split, thereby producing much finer fibers. Feeding the two components directly to the spinneret orifice produces side-by-side bico fibers and then they are combined near the orifice. Side-by-side fiber production can produce self-bulking and self-crimping fibers. Also “splittable” fibers forming fine filaments of 0.2 - 0.5 denier per filament are produced using side-by-side technology. Figure 1 shows different cross sections of side-by-side fibers.

2. Sheath/Core fibers:

Common sheath/core combinations are PE/PP, PE/PET, Co-PET/PET and PA/PET. The sheath polymer possesses the desired aesthetic or low temperature bonding properties and contains additives and colorants. The core polymer can either be a recycled polymer or an electrically conductive material. The sheath/core cross section is useful for applications where surface properties such as luster and dyeability and core properties such as strength is needed. Figure 2 shows some of the cross sections in which sheath/core fibers can be produced. Figure 3 shows a photomicrograph of cross section of one particular sheath PE/core PP fiber.

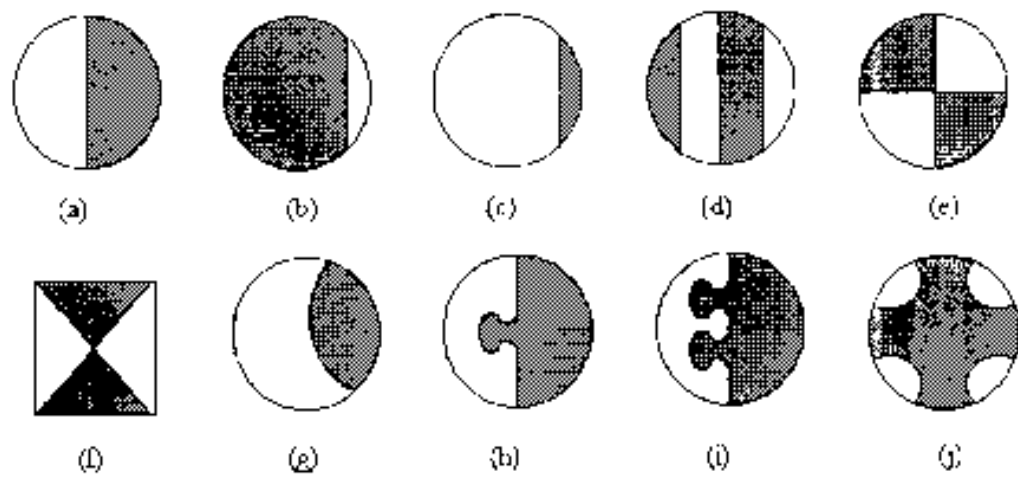


Figure 1. Cross sections of side-by-side bicomponent fibers

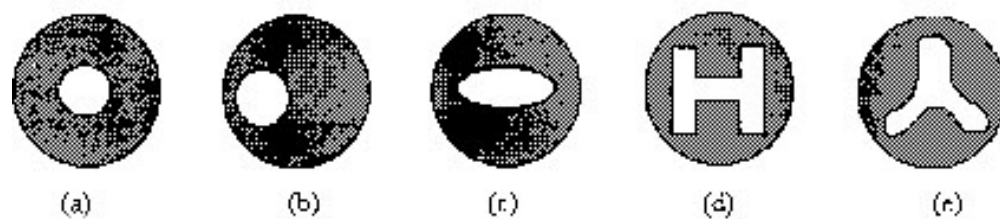


Figure 2. Cross sections of sheath-core bicomponent fibers

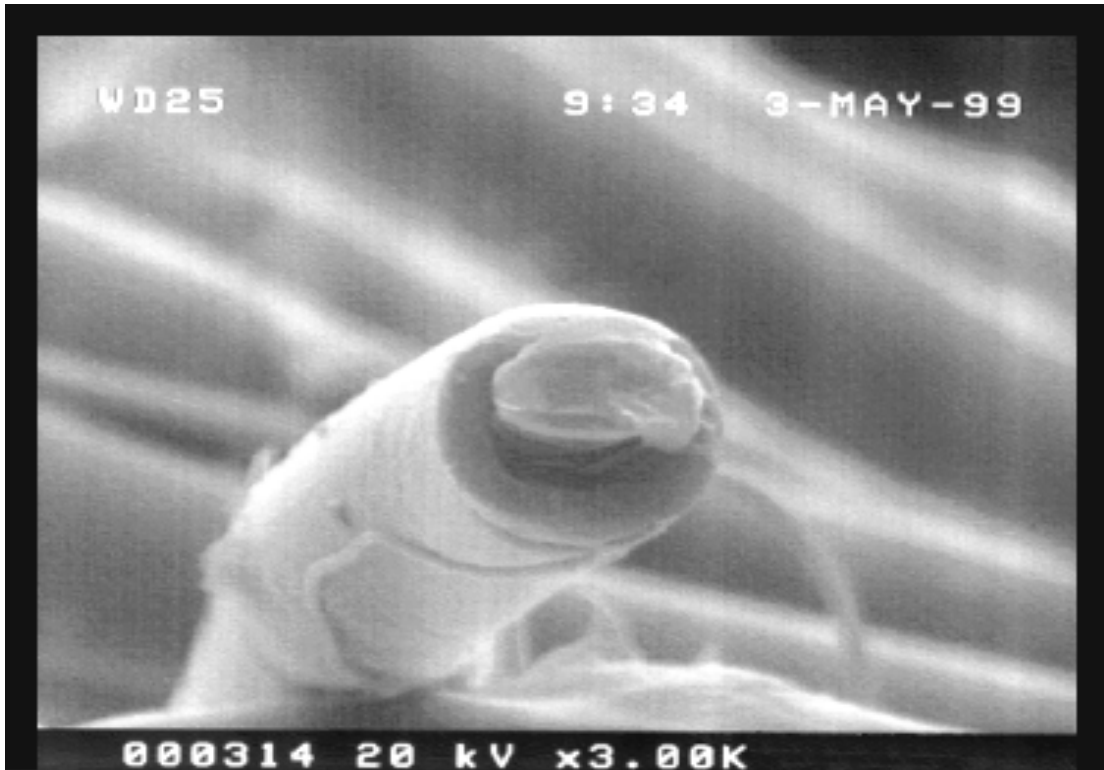


Figure 3. PE sheath/PP core [Reference: HILLS, Inc., 7785 Ellis Road, W. Melbourne, FL 32904]

3. Tipped fibers:

These are used to produce special aesthetics and other properties.

The above cross sections can be produced in any of the following geometries. Round cross-section, trilobal cross-section, sixteen segment pie, sixteen segment hollow pie and islands-in-a-sea. The proportions of the components in the bico fiber can vary from 10/90 to 80/20 depending on the applications and the polymers that are used.

Many bico fiber producers use PE as a sheath because it has excellent softness and has a low bonding temperature and is used as a binder material in fiber blends and PP as a core material as it has high strength. In addition to this, because of its low melting temperature PE allows the process to run at high speeds. Bicomponent fabrics made

of PP/PE offer greater softness and greater ease of bonding to films or SB nonwovens composed of PP. The following are some of the applications:

1. Nonwoven fabrics for diapers, feminine care, and adult incontinence products (Top sheet, backsheet, leg cuffs, elastic waistband, transfer layers).
2. Air-laid nonwoven structures (Absorbent core, wet wipes).
3. Spunlaced nonwoven products (wet wipes, medical disposable textiles, filtration products).

In the present research, bico PE/PP webs were produced on Reicofil® Bicomponent MB line. “The line, developed by Reifenhäuser GmbH & Co. of Troisdorf, Germany, represents a leading innovation in bico MB technology. Resin melt is delivered via two extruders, each with 50-kg/hr capacity. The hot air system capacity is 1,400 m³/hr with a maximum temperature of 350°C. The MB die is oriented vertically with a die-to-collector distance (DCD) range of 60 to 600 mm. The roll width is 24 inches untrimmed and 20 inches trimmed” [18]. The 100% PP and bico PE/PP webs with good basis weight and uniformity were produced on the new line with an air gap of 0.8 mm and a die tip set back of 1.0 mm. The polymers used were PP (Exxon Grade PP 3155) and PE (Dow Chemical 150 MI Linear Low Density Polyethylene (LLDPE)). The MB webs produced have side-by-side configuration of PE/PP fibers. PP MB webs comprise over 90% of the total MB production because of its low cost, ease of processing, good mechanical properties, lack of heat shrinkage, chemical inertness and ability to be drawn into fine fibers. As noted above, the PE allows for thermal bonding at lower temperatures or greater speed thereby producing softer bonded structures. PE

also has an inherently softer hand than does PP. MB webs have the following general properties.

1. Random fiber orientation.
2. Lower to moderate web strength; strength mainly due to mechanical entanglement and frictional forces.
3. Generally high opacity (having a high cover factor).
4. Fiber diameter ranges from 0.5 to 9 μm , but typically from 2-4 μm .
5. Basis weight ranges from 5-300 g/m^2 , but typically 10-50 g/m^2 .
6. Micro fibers provide a high surface area for good insulation and filtration characteristics.
7. Most MB webs are layered or shingled in structure even if produced from one die tip and the number of layers increases with basis weight.

SM and SMS Laminates: Joining SB and MB webs together for the final laminated web to attain the optimum properties of high strength of SB and barrier and filtration properties of MB webs produce SM and SMS laminates. The composite structure of the SB/MB/SB (SMS) and SB/MB (SM) are the most popular examples of the composite structures. These composite structures have been tremendously successful as they can be engineered to high strength products. SB and MB spinning beams are placed on the same machine in a configuration to facilitate the production of SM and SMS laminates. Some of the patents that are related to the present study are listed here with the description of methods and novel ideas that are present in them. Nonwoven SM laminates having higher tensile strength are disclosed in Brock et al U.S. Patent

No 4,041,203, which is assigned to Kimberly-Clark Corporation. The bulk, barrier and air permeability of non-woven laminates were identified and it was noted they were useful in protective clothing [22]. A SMS laminate produced and sold under the trade name KLEENGUARD® by Kimberly-Clark Corporation comprises of an internal layer of MB fibers sandwiched between two layers of SB filaments of PP and PE [28]. The study of three-layered non-woven laminate with two exterior layers of SB PP and an internal layer of mixture of MB PE and PP by Brock et al. [30] was found useful in producing strong laminates with good barrier properties.

Apparatus for the manufacture of nonwoven webs and laminates including means to move the spinning assembly. [32]

A multi-station line consisting of at least one SB die assembly and at least one MB die assembly produces SM and SMS laminates. Each station includes

a) A melt spinning die which can be selectively equipped with a SB die insert or a MB die insert and

b) A moveable support structure for adjusting the proper die-to-collector (DCD) distance, depending on SB or MB mode of operation. The multi-station line permits the selective manufacture of a variety of SM or SMS laminates, including SMMS laminates. The layers may be bonded together by compaction or by calendering and exhibit outstanding strength properties, energy absorption, tensile strength and tear resistance, and yet possess a soft, flexible hand. The SMS structure is typically made inline wherein (A) SB filaments are laid on a moveable collector forming a first layer, (B) MB filaments are deposited on the first layer, and finally (C) a second layer of SB filaments is deposited on the top of the MB layer. The three-layered structure then can

be bonded together. The inline operation is restricted to manufacturing only one SM and SMS laminates. However, the use of bico and/or blend fibers requires more complex equipment than required for monofilaments, and can also require additional processing steps. The Bico MB fibers can either be sheath/core or side-by-side. This research was focused on SM and SMS composites made from side-by-side bico fiber PP/PE MB webs. Preliminary work has shown that SM and SMS had a softer hand and lower flexural rigidity than did laminates made from 100% PP MB [5].

The present study is based on lamination of different webs produced from SB and MB processes with the concentration on studying the effect of % of PP on the laminates produced. A statistical response surface design employed for the design of the experiments to be conducted based on the processing parameters. The processing parameters, mechanical properties together with the proportion of PP and PE were studied for optimizing the processing parameters to achieve optimum values for basis weight, air permeability, hydrostatic head and bending length. The Bico MB PP webs and 100% PP, PE MB webs were produced on the 24-inch Reicofil MB line and were unrolled during spinning of the SB filaments. Experimental studies were also made to determine if better bonding and performance properties resulted from the MB being on top against the patterned calender roll versus the MB against the smooth calender roll.

CHAPTER III

EXPERIMENTAL PROCEDURES

The MB webs of bicomponent (bico) fibers were produced on the 24-inch Reicofil® MB line at TANDEC. Polypropylene (PP) and Polyethylene (PE) were used as the components of the MB fibers. In order to study the effect of the relative proportions of PP and PE in the bico MB pairs on the performance properties of the laminates, MB webs containing following percentages with two different basis weights (10g/m^2 and 20 g/m^2) were produced: 25%PP/75%PE, 50%PP/50%PE, 75%PP/25%PE, 100%PP and 100%PE. The average fiber diameters of the MB webs are shown in Table 1.

Exxon PP 3155 (35 MFR) was used to produce the SB webs. Exxon PP 3546G (1200 MFR) and PE (Dow Chemical 150 MI Linear Low Density Polyethylene (LLDPE)) were used in the production of bico MB webs for the manufacture of SM and SMS laminates. The processing parameters of both MB and SB webs produced are listed in Table 2. The SB web was produced in line and bonded together with the MB webs to produce SM laminates at three different thermal bonding temperatures and three different calender nip pressures. Sandwiching the MB web between the SB webs produced SMS laminates. For the production of SMS laminates, a slightly pre-bonded 100% PP SB nonwoven web was produced on the 1-meter Reicofil® 2 SB line and was placed on the smooth roll side. The MB webs were laid over the pre-bonded SB webs and the top SB web was produced on-line and laid over the MB layers just prior to the thermal bonding. The main processing parameters listed in Table 2.

Table 1. Average fiber diameters of the MB fibers

Meltblown webs	Fiber diameter (μm)
100% PE/0% PP (10 gsm)	3.10
100% PE/0% PP (20 gsm)	3.17
25% PP/75% PE (10 gsm)	2.48
25% PP/75% PE (20 gsm)	2.85
50% PP/50% PE (20 gsm)	2.52
75% PP/25% PE (10 gsm)	2.59
75% PP/25% PE (20 gsm)	2.54

Table 2. Primary processing parameters of the SB and MB webs

WEBS	PRIMARY PROCESSING PARAMETERS				
Spunbond	Web Forming			Bonding	
Pre-bonded PP SB	Die melt temp. 445°F; Cooling Air temp. 66°F; Quench Chamber Pressure. 538Pa; Spin Belt Speed. 60.3 m/min; Suction Air speed. 1484 RPM			Upper Roll-247°F; Lower Roll-242°F; Nip Pressure-97PLI	
In-line PP SB				See “Experimental Design”	
Melt Blown	Die Temp °F	Through put g/hole/min	Air Rate SCFM	Air Temp °F	DCD in
75%PP/25%PE	500	0.546	350	480	6
50%PP/25%PE	500	0.557	348	479	6
25%PP/75%PE	500	0.546	348	478	6
100% PP	520	.546	298	514	6

The web was characterized according to current ASTM and INDA test methods for the following properties and their values are shown in Table 3. In the case of both SM and SMS laminates, the SB webs were produced in line under the same conditions in terms of polymer throughput, die melt temperature, cooling air temperature, quench air chamber pressure, suction speed and spin belt speed as shown in Table 2.

Production of nonwoven composites:

The SM composites were produced in two ways without utilizing the lightly pre-bonded SB web. In one of them, the SB filament is formed directly onto the MB web so that the SB web was in contact with the upper diamond patterned calender roll with 14.7% raised area as shown in Figure 4. In the other procedure, the MB web was unwound onto the newly formed SB filament web before passing through the calender, which allowed the MB web in contact with the upper calender roll as shown in Figure 5. The processing conditions were maintained the same for both the SM and MS laminates. In Figure 6, a process that closely simulates the production of SMS is depicted in which the lightly pre-bonded SB component is on the bottom side. The MB layer is unwound so that the SB filament web is formed directly onto the MB web. The three-layered SMS laminate is passed through the calender allowing the lightly pre-bonded SB web in contact with the lower smooth calender roll and the in-line SB web in contact with the upper patterned calender roll. The production speed for both SM and SMS was set at 60.3 m/min in order to keep the same basis weight of in-line SB web as that of lightly pre-bonded SM web; whereas, the bonding temperature and pressure varied according to the experimental design.

Table 3: Mechanical properties of the 100% PP SB produced on 1-meter Reicofil® SB line

Mechanical Property	Value
Basis weight	11.6 g/m ²
Thickness	0.163 mm
Bending length	2.15 cm
Bursting strength	8.5 lb/in ²
Air permeability	170 ft ³ /ft ² /min
Hydrostatic head	11.5 cm
Peak load	0.48 kg
Elongation-at-break	102 %

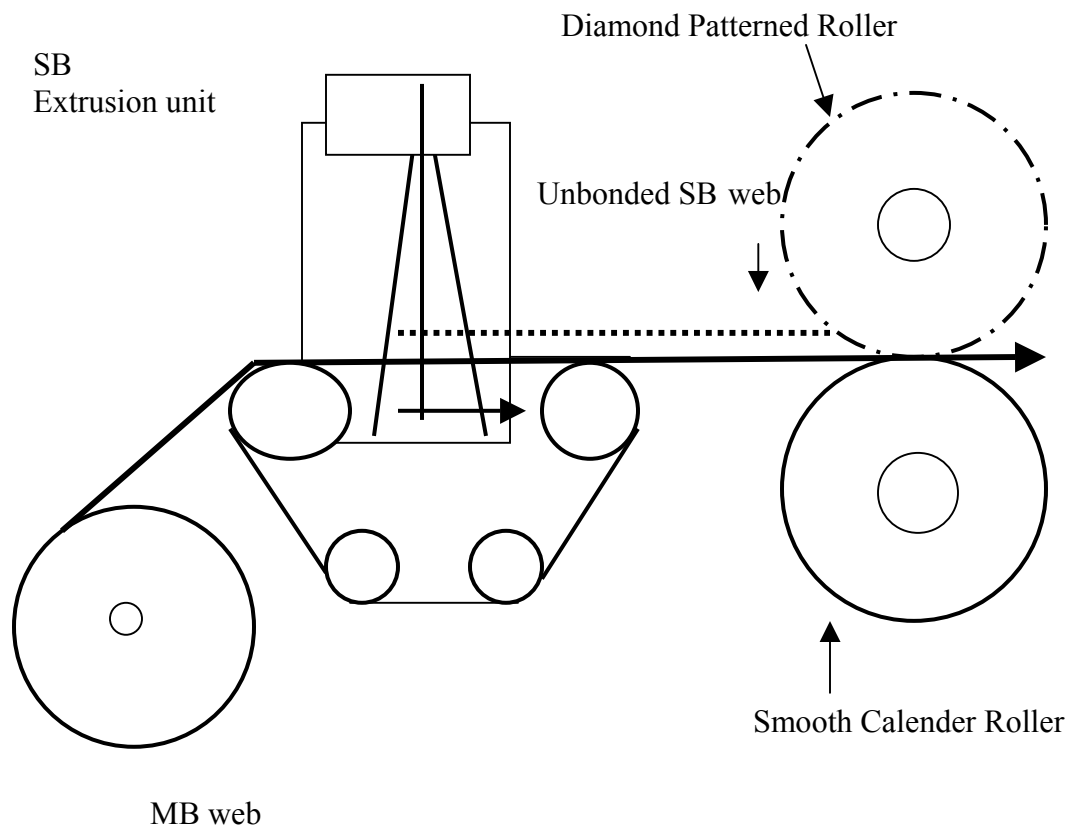


Figure 4. Production of SM Laminates by unwinding a MB web on the bottom side of the unbonded SB web produced from the SB extrusion unit

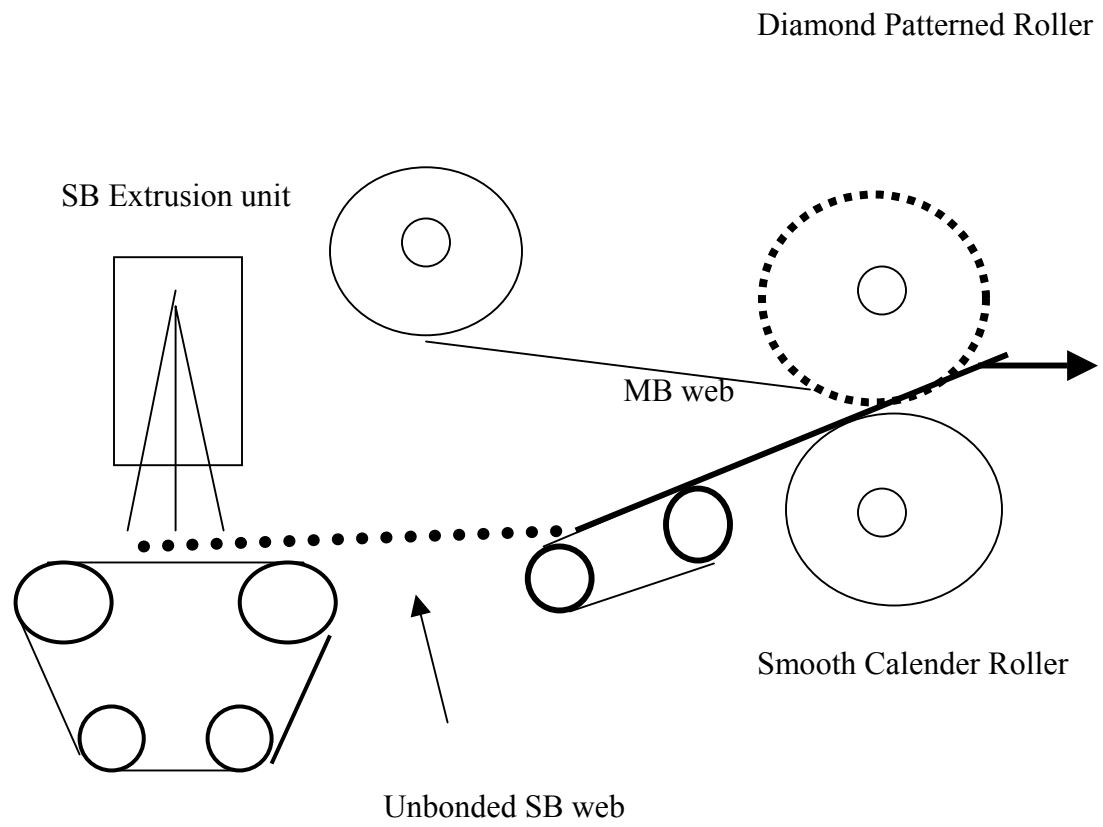


Figure 5. Process for production of SM by unwinding a MB web on top of an unbonded SB PP web drawn from the extrusion unit

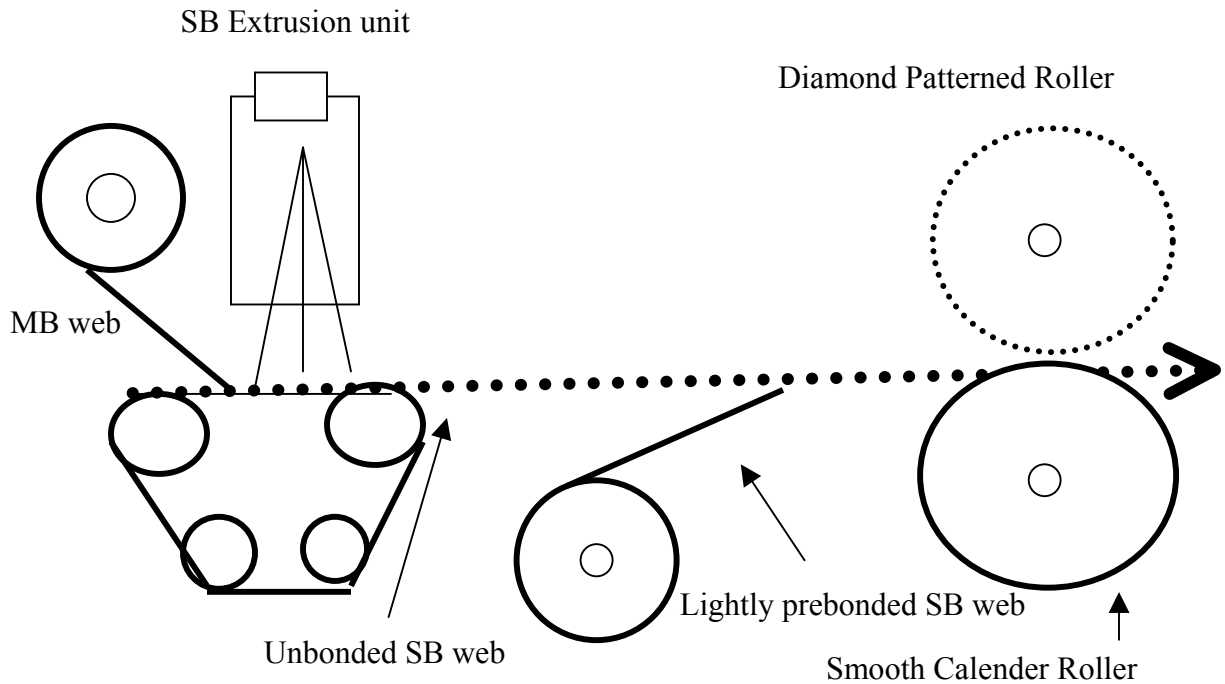


Figure 6. Production line utilizing 1.0 m SB line simulating the production of SMS laminates

Experimental Design and Characterization:

Four factors; thermal bonding temperature, calender nip pressure, percentage of PP in the bico MB web and bico basis weight were particularly studied in this research. The Response Surface Method (RSM) was employed to investigate the effects of these factors on the SM and SMS end-use properties. Two factors had three levels as listed in Table 4. Both SM and SMS were produced with two types of MB webs, 10 gm/m²

and 20gm/m² respectively. Therefore the total number of runs in this research was 90. After the production; the composites, as well as the single SB and MB webs, were characterized according to the current ASTM and INDA test methods for the following physical and performance properties: basis weight, thickness, bulk density, hydrostatic head, air permeability and flexural rigidity which is calculated from the bending length. With the 100% PP, we were not able to produce samples at a bonding temperature of 300°F, and the final number of samples produced were different from what we had expected to produce, though the results of the samples were analyzed based on the four factors that were shown in Table 4.

Table 4: Factors and levels investigated

Factor	Label	Low		Middle		High	
Temp	Bonding temperature of top roll in °F	250		275		300	
Pressure	Bonding pressure in pound/linear inch	240		255		270	
PP	Percentage of PP in bico web	0	25	50	75	100	
Bico weight	Basis weight of the bicomponent PP/PE MB web	10			20		

Mechanical properties:

SM and SMS webs thus produced were tested for mechanical properties according to the INDA standards. The test methods are described below.

Basis weight [IST 130.2 (98)]

The mass per unit area of nonwovens is expressed in grams per square meter. It is determined by cutting test pieces of 10”x10” from a nonwoven web and weighing them using the Mettler AE 240 tester. The average of the fabrics weighed is converted into Grams per Meter Square. 10 samples were taken and their average calculated.

Bursting strength [IST 30.1 & ASTM D3786-87]

A specimen of the fabric is clamped over an expandable diaphragm. The diaphragm is expanded by fluid pressure to the point of specimen rupture. The difference between the total pressures required to rupture the specimen and the pressure required to inflate the diaphragm is noted down as bursting strength of the web. 5 samples were tested for each sample and their average is the bursting strength of that particular sample.

The instrument used was “The Mullen Tester (Serial No 72 c 744) manufactured by B.F. Perkins”.

Hydrostatic head [IST 80.6 (98)]

A nonwoven fabric specimen is mounted on the test head reservoir. The specimen is subjected to a standardized water pressure, which is increased at a constant rate until leakage appears on the specimen. Water pressure is measured as the hydrostatic head height is reached at the first sign leakage in three separate areas on the specimen. A higher value indicates greater resistance to water penetration. The average of five

samples was calculated using TEXTEST FX 3000 hydrostatic head tester.

Air permeability [IST 70.1 & ASTM D 737-96].

The rate of airflow passing perpendicularly through a known area of fabric is adjusted to obtain a prescribed air pressure differential between the two fabric surfaces. From this rate of airflow, the air permeability of the fabric is calculated. The average air permeability value from 5 samples is determined using the TEXTEST FX 3300 Air permeability tester.

Flexural rigidity [IST 90.1 (01) ASTM D 5732-95]

A rectangular specimen (10" x 1") is slid at a specified rate in a direction parallel to its long dimension, so that its leading edge projects from the edge of a horizontal surface.

The length of the overhang is measured when the tip of the specimen is depressed under its own weight to the point where the line joining the top to the edge of the platform makes a 0.785 rad (41.5°) angle with the horizontal. The stiffer the web or the fabric, the longer it takes to bend, thus the higher number indicates a stiffer fabric.

Four rectangular samples were cut with the long dimension parallel to the machine direction for each web and the four sides of each sample are tested using FRL

Cantilever bending tester [Made by Testing Machines Inc. Amityville, NY] and the average is calculated which gives the bending length of the particular web. Flexural rigidity of the web is calculated from the following formula

$$G = 9.809 \times 10^6 \times M \times C^3$$

Where G = Flexural rigidity, $\mu\text{N.m}$ and

M = Fabric Mass per unit area g/m^2 and C = Bending length in mm

Thickness [IST 20.1 & ASTM D5729-97]

Thickness of the nonwoven web is determined by observing the linear distance that a movable plane is displaced from a parallel surface by the specimen while the specimen is under pressure. The thickness of 5 samples was found using Thickness tester (Made by TMI, Amityville, NY) and the average of them gives the thickness of the web.

Tensile testing [IST 110.4 & ASTM D 5035-95]

A test specimen is clamped in a tensile testing machine and a force applied to the specimen until the specimen breaks. Values for breaking force and elongation of the test specimen are obtained from machine scales, dials, autographic recording charts, or a computer interfaced with the testing machine. Here 5 samples of each web are tested and the values of peak load and elongation break of the web were calculated from the machinery readings. United Testing Systems, Inc; Huntington Beach, California, manufactures the machine (Model No SSTM.1.E.PC, Serial No 692522) used for tensile testing.

CHAPTER IV

RESULTS & DISCUSSIONS

Comparison of properties of thermally bonded SM and SMS composites:

SM composites are made by a lamination process in which SB filaments formed from the spunbond line are laid on the MB web and the SB web is in contact with the upper diamond patterned roller or vice versa. The MB webs may be in contact with the upper patterned roll and the other web is in contact with the heated smooth steel roller. The MB webs were either mono components of PP or PE or bicomponents (bico) of 25% PP/75% PE, 50% PP/50% PE, 75% PP/25% PE and 10g/m² and 20 g/m² respectively. Figures 7 and 8 show the comparison of SM and SMS in AP and HH. The two composites exhibit approximately the same air permeability with the same basis weight MB webs, although SM shows slightly higher air permeability. As would be expected, composite containing thicker MB webs (20g/m²) has much lower AP. Under the bonding conditions of 300° F/270 PLI and 60.3-m/min surface speed of calender roller, the difference in AP is negligible. This may be due to the pinhole formation around the bond area in the laminates containing lower basis weight bico MB, at the higher temperatures. For hydrostatic head (HH), equal or higher values were observed for SMS composites as shown in Figure 8. In the case of HH, slightly higher values were obtained for a bonding temperature of 250°F and the bonding pressure of 240 PLI to 270 PLI. However, SM composites produced at 300°F/240-270PLI exhibit lower HH, which may be due to pinhole near or in the bonding points. This situation seems

less significant for SMS with 20 g/m² with a ratio of 75%PP/ 25%PE MB web. These results show that the PE component in the MB leads for responsiveness to the bonding conditions, which will be an advantage with higher basis weight of MB in the SM or SMS composite to achieve greater barrier properties. Figure 9 shows the flexural rigidity (FR) of the composites produced under different processing conditions. In general, SMS composites have higher FR than the SM composites do and those containing heavier MB webs are relatively stiffer than those having lighter MB webs. Under higher bonding temperature (300°F), the effect of bonding pressure on FR is significant. Both 10 and 20 g/m² MB containing SM composites possess notably lower FR at higher bonding pressure (and the same bonding temperature of 300°F). Therefore the effects of bonding conditions on FR depend on the layers of the laminate and the basis weight of the layers. Combining data in Figures 7, 8 and 9 of 300°F/270 PLI, one may conclude that high bonding temperature and pressure would generate thinner bonding points with possible pinholes at edges, which leads to lower FR and HH but higher AP. SMS composites containing heavier basis weight MB webs produced relatively thick bonding points without pinholes; therefore, their FR and HH values are high, and AP values are low. The tensile strength of the SM and SMS composites produced under varying bonding conditions were presented in Figure 10. The bico MB webs in these composites are 50%PP/50%PE, with a basis weight of 20 g/m². It is found that the tensile strength of these SMS composites remains nearly constant, because the two SB layers (accounting for ~50% weight of SMS) determine the tensile property.

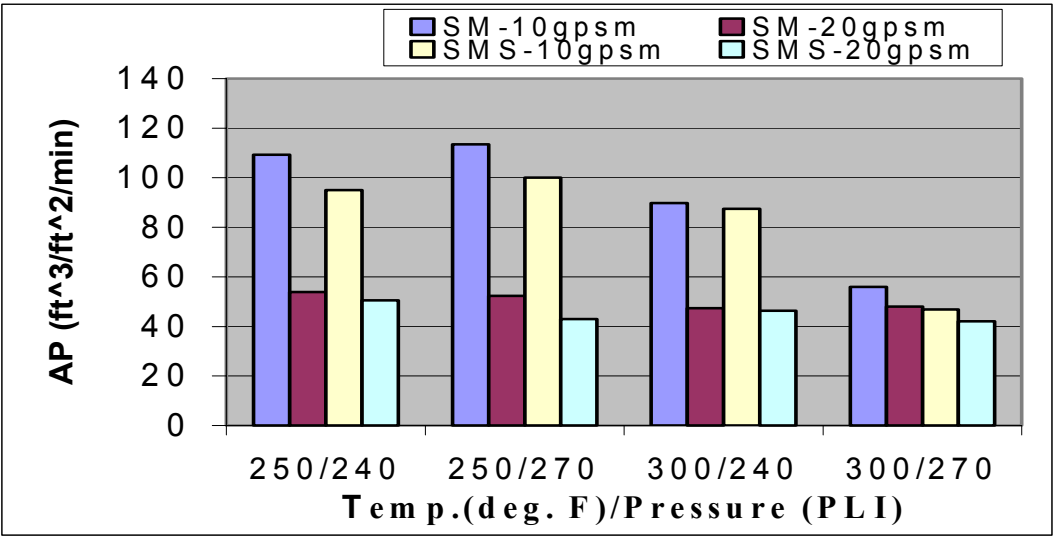


Figure 7. Comparison of SM and SMS Composites in Air permeability (75%PP/25%PE)

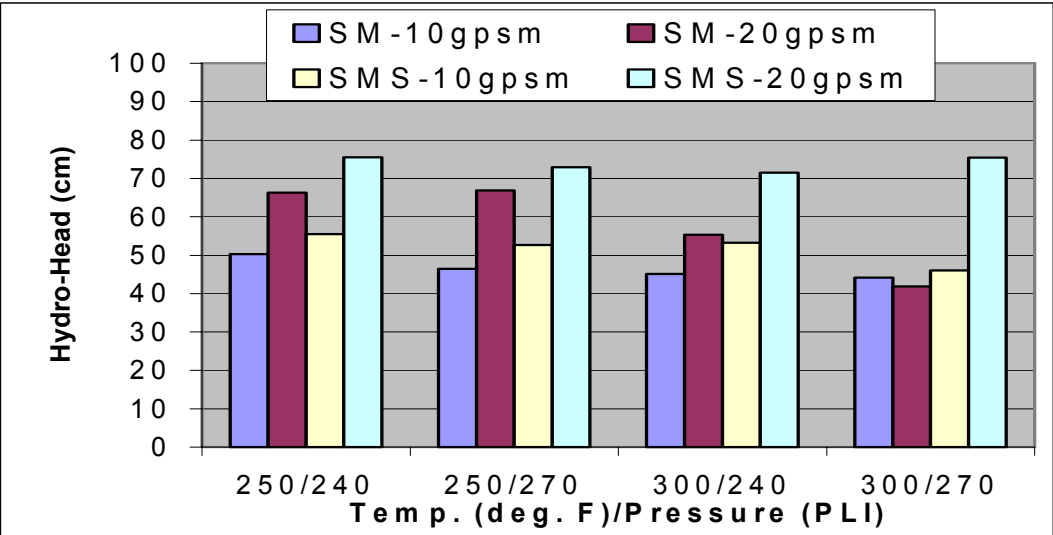


Figure 8. Comparison of SM and SMS Composites in Hydrostatic Head (75%PP/25%PE)

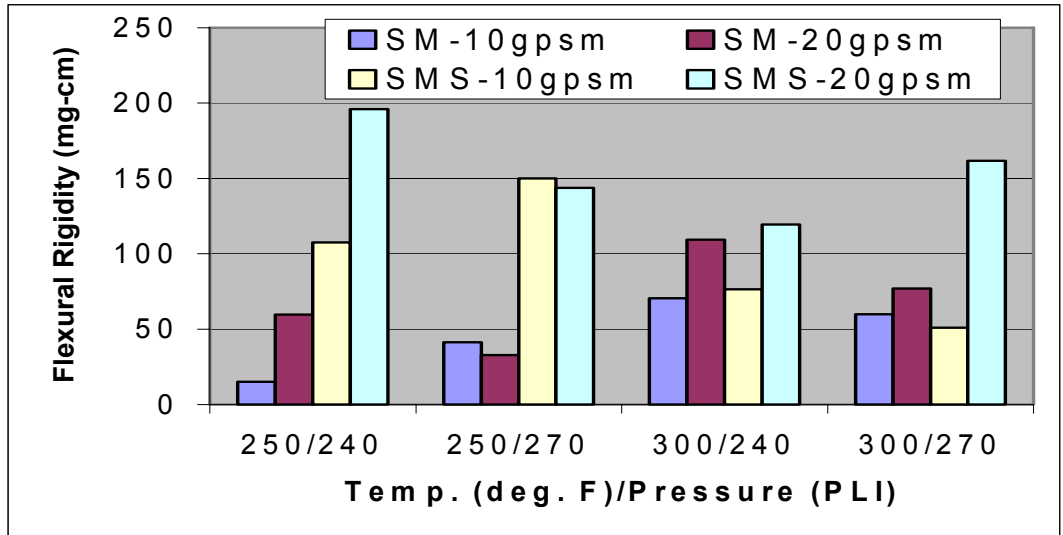


Figure 9. Comparison of SM and SMS composites in Flexural Rigidity (75%PP/25%PE)

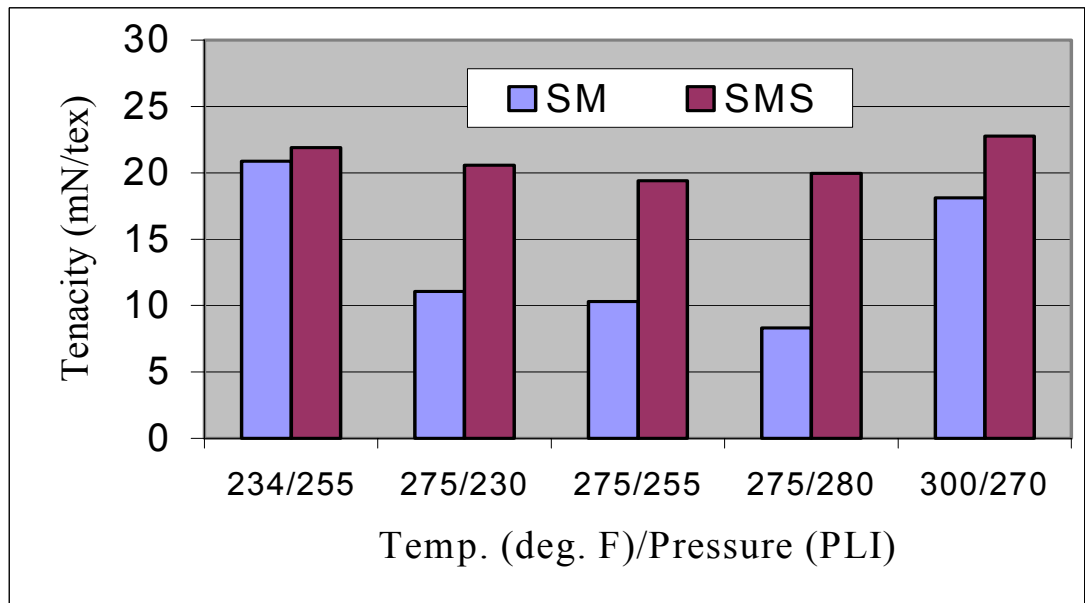


Figure 10. Comparison of SM and SMS Composites in Tensile Strength (50%PP/50%PE, 20gm/m²)

Comparison of properties of SM produced with MB-on-top and MB-in-bottom:

As described in the experimental production section, SM composites were produced with the MB web on the top and bottom side of the SB filament web, respectively. The testing results in HH and AP were plotted in Figures 11 and 12. For those containing a 20g/m² MB web, the SM composites produced in the two different ways exhibit essentially the same AP at low bonding temperature. Notable difference in AP is obtained with higher values of MB-on-top SM at high bonding temperature. This difference is also observed as the percentage of PP in the bico MB web varies. The results of HH seem more sensitive to material (MB webs) and bonding conditions, as shown in Figure 12. At low bonding temperature, the two production procedures do not affect the HH values notably for the 75% PP/25% PE MB containing SM; whereas it is significant for 25% PP/75% PE containing SM, because the main component PE has lower melting point and is more sensitive to pressure. At high bonding temperature (300°F), SM laminates with 25% PP/75% PE MB on top against the upper patterned roller was seemingly over-bonded, which results in lower HH. One would not be surprised by this result because PE is the predominant component in that bico MB web. Under the same conditions, higher HH values were obtained from the SM composite with 75% PP MB on top. The apparent effect of the presence of % of PE, which has a lower melting point than that of PP, on over-bonding and in turn on the mechanical properties of the samples was studied using SEM images. SEM images of the bonding points of the samples reveal that at the higher bonding temperatures of 300°F over-bonding took place which may have resulted in the drop of HH and FR values and a rise in AP values. Figure 13 shows the SEM image of the

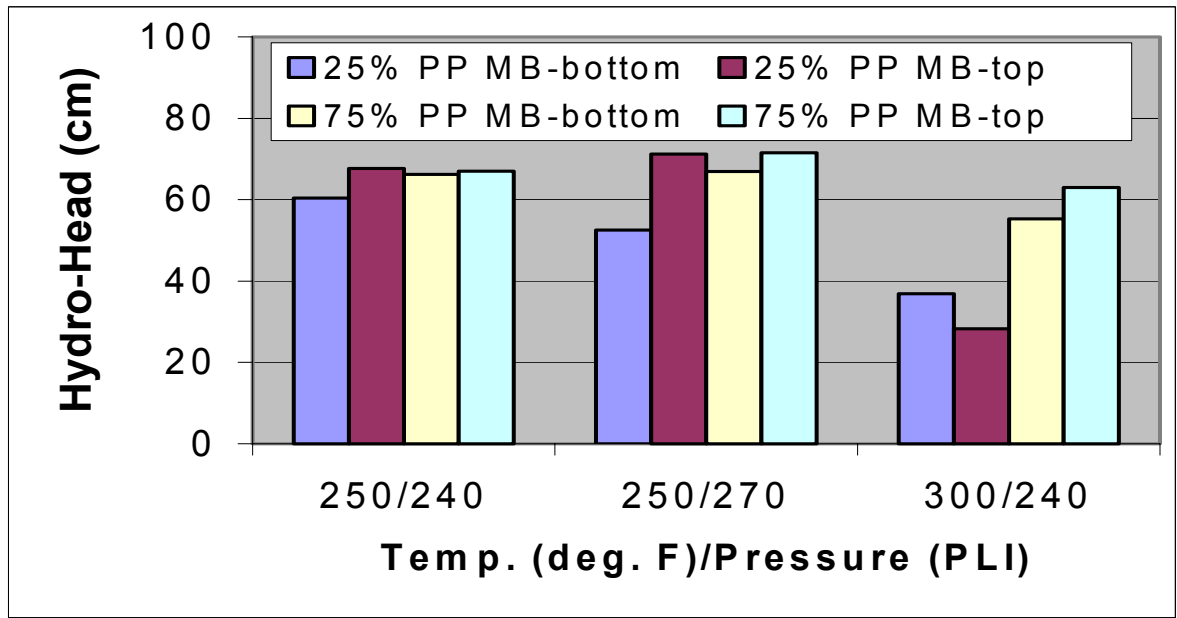


Figure 11. Air permeability of SM composites with MB-on-top and MB-in-bottom (MB 20gm/m²)

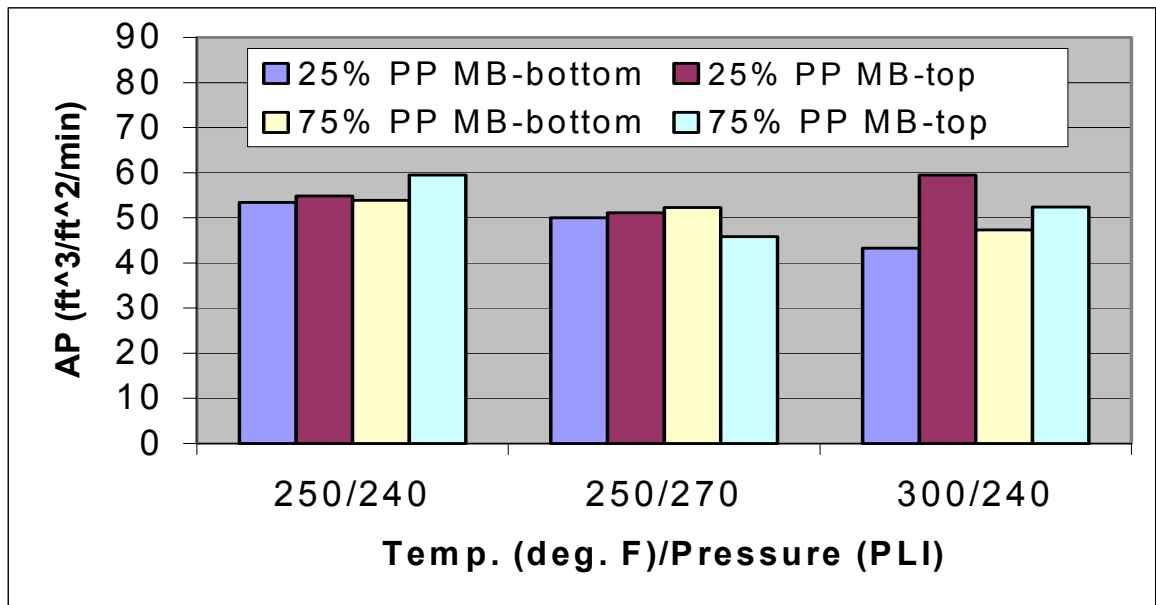


Figure 12. Hydrostatic head of SM composites with MB-on-top and MB-in-bottom (MB 20gm/m²)

SM sample containing 25 %PP/75%PE as bico MB fiber bonded at 300°F. The image shows good bonding with MB and SB fibers being fused together. The flattened SB filaments are still visible in the bond area. However, the SB filaments protruded from the bond area. No breaking of the fibers at the bond points is apparent. Figure 14 shows the SEM image of SMS sample containing 25% PP/75% PE bico MB web bonded at 300°F but at a lower PLI of 240. It shows almost the same degree of the thermal fusion of the SB and MB fibers in the bonded area, as does the sample in Figure 13, which was essentially the same except the PLI was lower at 240. Thus, changing the bonding temperature appears to have a much larger effect on bonding than does pressure. Figure 15 shows an image of SMS laminate containing 100% PP MB fiber. It appears completely bonded. The resultant web may appear stiffer and more brittle structure. It has low HH values, which may be due to pinhole formation.

Effects of PP percentage in the bico PP/PE MB web on laminates:

The percentage of PP in bico fiber MB web affects the physical performance of SM and SMS composites as shown in Figure 16. This effect varies with the production conditions therefore and is not an independent factor. In figures 16 and 17, the SM and SMS composites were produced at a bonding temperature between 250° F and 270° F and a bonding pressure between 240 PLI and 255 PLI with the laminates. In these ranges, barrier properties i.e. hydrostatic head and air permeability do not change with the bonding temperature and pressure; therefore, the effect of PP percentage can be investigated using the available data. The HH and AP values reach a maximum at reach a maximum at 75%PP/25%PE.



Figure 13. SEM image of SM sample (300°F, 270 PLI, 25%PP/75%PE)

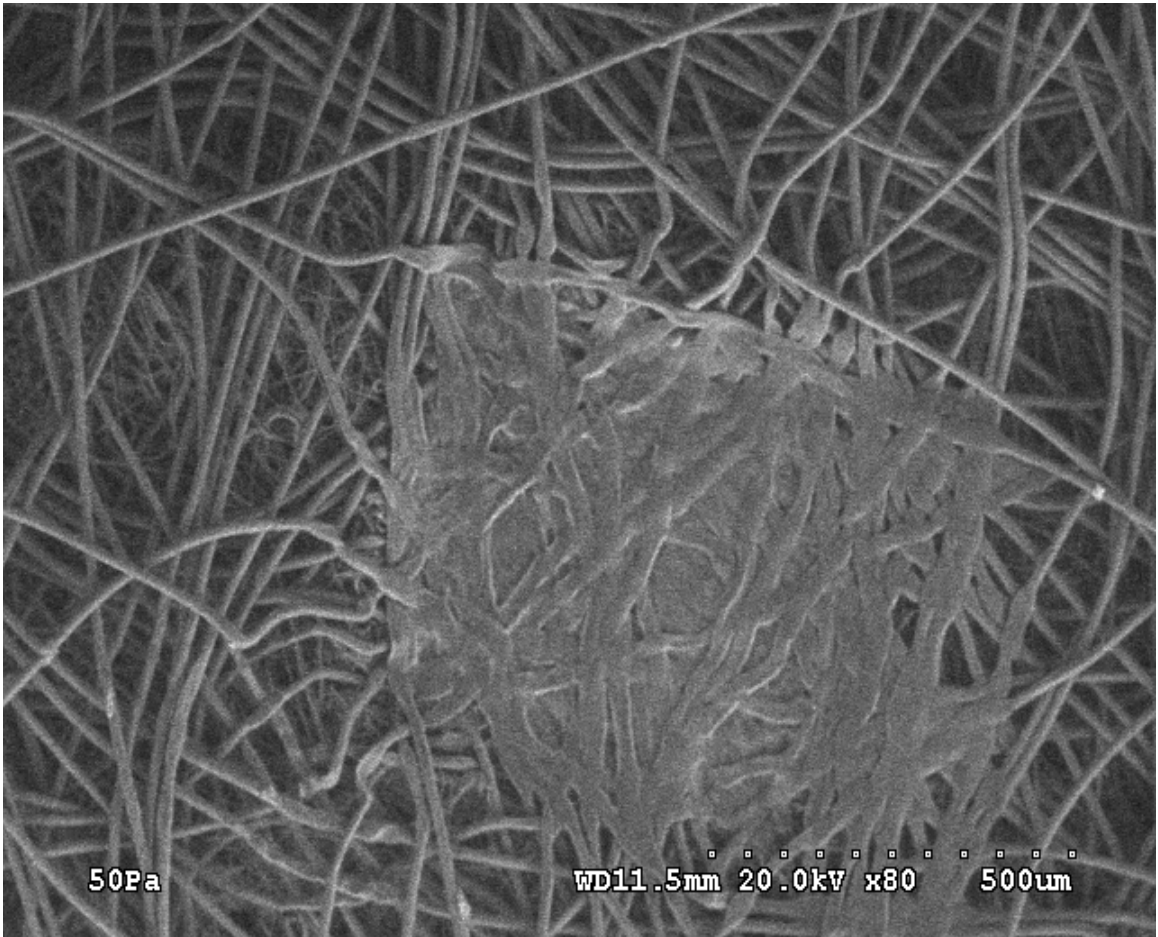


Figure 14. SEM image of SMS sample (300°F, 240 PLI, 25%PP)

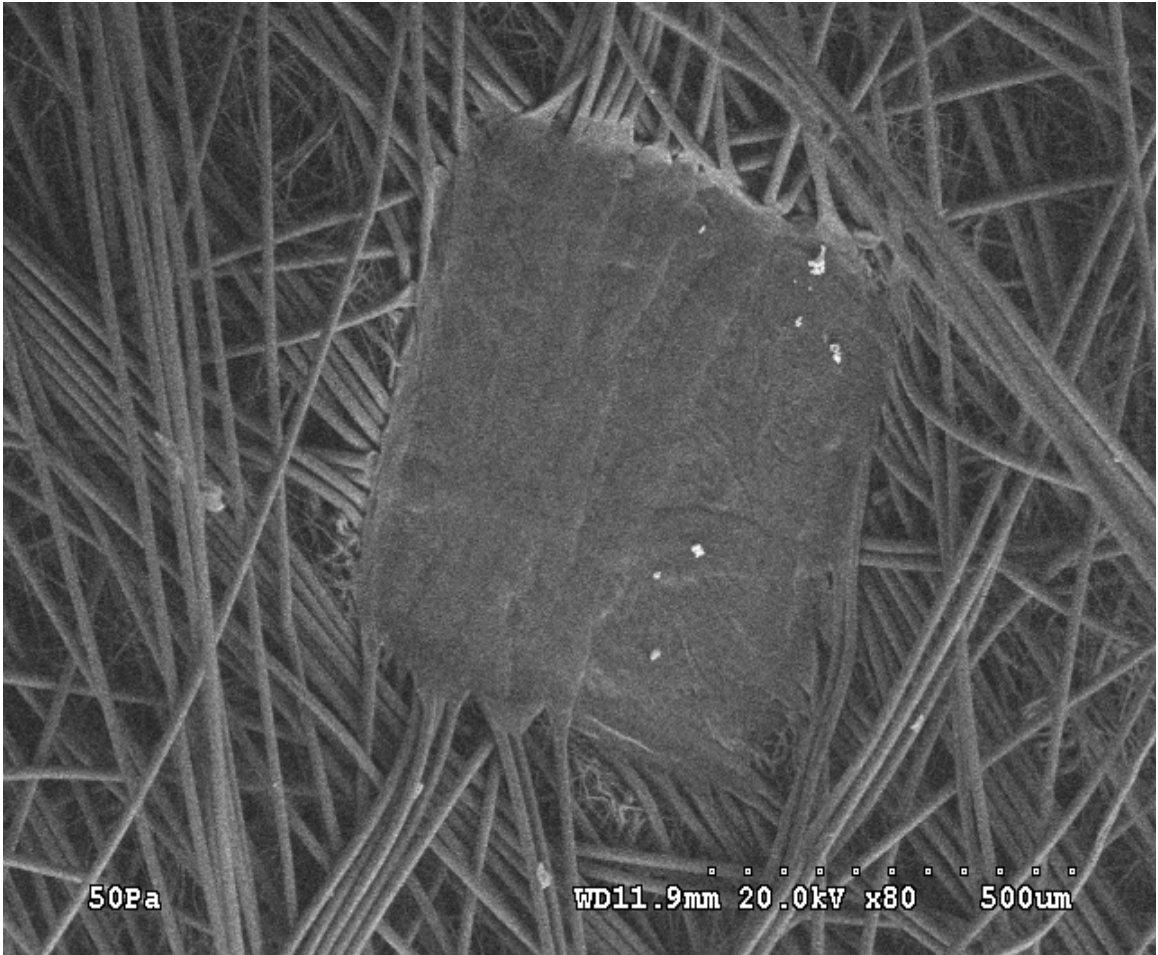


Figure 15. SEM image of SMS sample containing 100% PP MB fiber (275°F, 260 PLI)

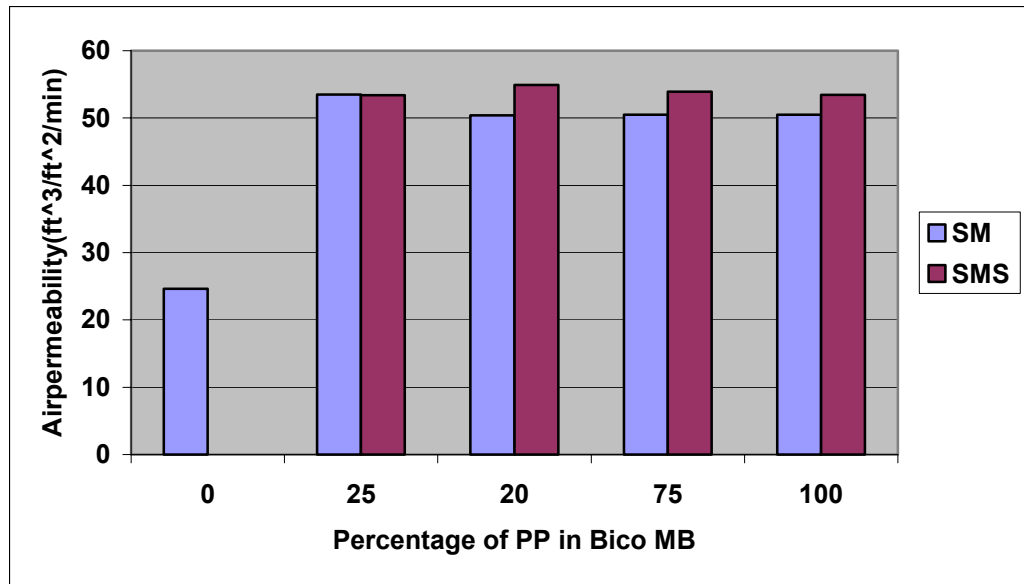


Figure 16. Effect of % PP in bico MB webs on Air permeability

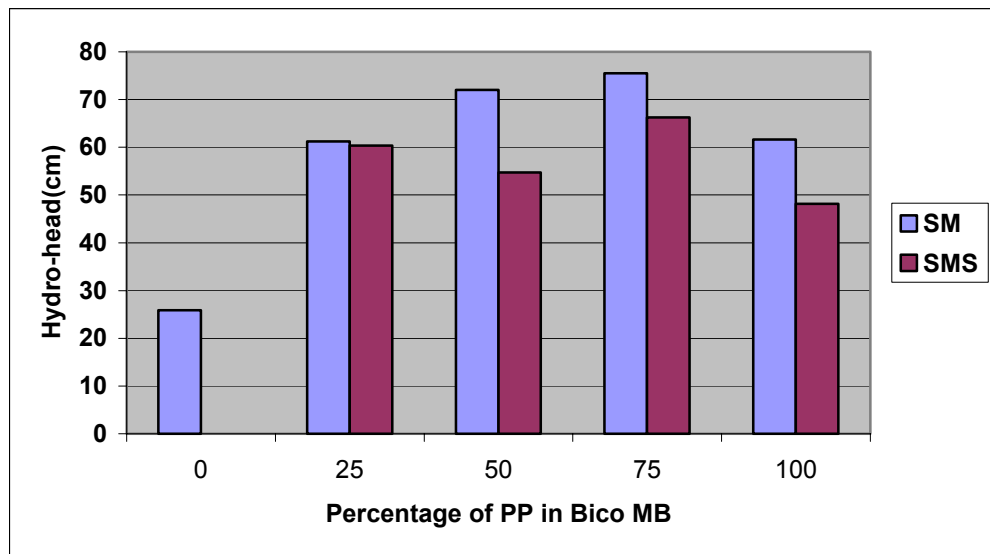


Figure 17. Effect of % PP in bico MB webs on Hydrostatic head

Statistical analysis:

The Response Surface Method (RSM) is a statistical technique for approaching a system to assess the effects of factors on the behavior of measurable quantity (Response). They are employed before, while and after a regression is performed on the data. The experiment must be designed before the regression analysis by inputting the factors that may have an effect on the desired/required responses. Optimization methods and model testing procedures are employed after the regression is performed. The subject of RSM enables us to gain a better understanding of the characteristics of the response system under study.

Factors: Factors are processing conditions or input variables whose values or setting can be controlled by the experimenter. If one changes the settings of the factors, the value of response variable changes as well. In this research the factors are bico web basis weight, bonding temperature, calender nip pressure and % PP in the bico PP/PE MB web, which have an effect on the properties of the laminates.

Response: The response variable is the measured quantity whose values depend on the levels of the factors. In this research hydrostatic head pressure (HH), air permeability (AP), flexural rigidity (FR) and tenacity are the responses, which are tested for all the samples produced with different combinations of the levels of the factors.

Its applications generally include

1. Showing how a particular response is affected by a set of variables over some specific region of interest.

2. Discovering the setting of factors that will give a product simultaneously satisfying specifications for a number of response
3. Exploring the space of the factor variables to define the maximum response and determine the nature of the maximum. [15]

The true value of the response corresponding to any particular combination of factor levels and in the absence of experimental error is denoted by η . Experimental error may result due to the production equipment, the testing equipment, the people who run the experiment, and the other miscellaneous errors.

The experimental runs were conducted using a central composite uniform precision technique in RSM to randomize the runs to minimize the variation within each temperature zone. Temperature was taken as a whole unit factor to minimize the production time of the laminates. Inserting the bico MB of varying proportions of PP and PE and the two basis weights of 10 and 20 g/m² produced laminates bonded at 250 °F and 240 pounds per linear inch (PLI). Only one MB roll of each proportion was used to produce the various combinations of laminates so as to reduce the variation due to between MB rolls. In other words laminates containing 25% PP were produced using one MB roll which has 25% PP/ 75% PE. With the treatment structure 3X3X2X5X2, the total number of laminates, which were planned, was 180. But the actual number of laminates produced were 90 due to technical problems and lack of 50%/50% PP/PE MB webs. The total number of laminates includes MS laminates, which were produced to investigate the effect of MB as top layer and MB as bottom layer of SM laminates. The experimental design for the laminates is shown in the Figure 18.

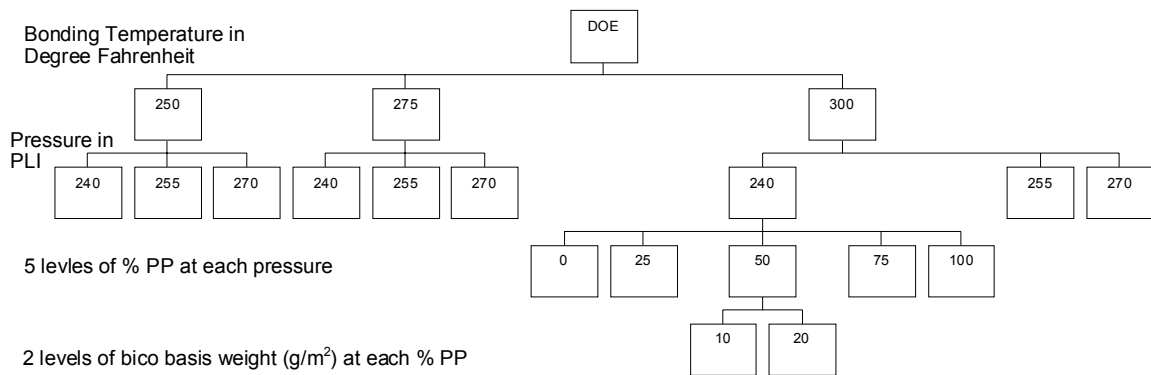


Figure 18: Treatment structure for preparation of laminates

100 % PE laminates were not taken into analysis, as enough data for that particular level of PP is not available.

Class Level Information

Class	Levels	Values
temp	3	250 275 300
press	3	240 255 270
pp	4	25 50 75 100
Weight	2	10 20
type	2	sm sms

SAS, statistical software was used to obtain correlations between the factors and responses and for analyzing the effect of factors on the responses. Contour plots and surface plots showing the variation of responses with 2 or more factors were drawn using the above software. General linear models (GLM) and ANOVA were performed using this software to analyze the data. Regression using the response surface model was used to investigate the effect of factors on responses.

Observing the GLM procedure for FR; the probability values are significant at 0.05 level for % PP, weight and type indicating that these independent variables have a significant effect on FR. Bonding temperature and calender nip pressure do not have significant effects on FR. % PP * weight, % PP * type and weight * type are the interaction terms that are significant for FR in addition to the main effects of % PP, bico and type of the laminate. Significant variables affecting the HH are temperature, % PP, weight and type of the laminate. The interaction terms that are significant are [temp * % PP], [temp * weight], [temp * type], [% PP * weight], [% PP * type], [weight * type]. AP was not affected by bonding temperature and calender nip pressure. Bico basis weight and type of the laminate have a significant effect on AP. The interaction term that is present in the analysis is weight*type. A comparison between temperatures 275 °F-300 °F is significant at 0.05 level. A comparison between % PP is significant at 25%-50%, 25%-75% and 25%-100% levels. As little as 25% PP and percentages more than 25 have modified the properties to a notable extent. Maintaining PP at 75 % and varying the bico basis weight has a significant effect on all of the properties. At the 100% PP level, both bico basis weight and type of the laminate, whether it is SM or SMS, has a significant effect. From the analysis it

is observed that the effect of bonding temperature and calender nip pressure on the laminates are not significant but the effect of % PP and bico basis weight have a significant effect on the various responses studied. % PP and bico basis weight has a strong correlation with the properties such as FR, HH and AP. SMS laminates containing three layers have good mechanical properties compared with the SM laminates containing two layers. Coefficient of determination (R^2) values is low for FR and AP, whereas for HH and tenacity the R^2 value indicates that the responses have correlation with the factors temperature, % PP and bico basis weight. Bonding pressure does not have a significant effect on the responses. A study of the correlation tables shows that HH and bico basis weight have a strong correlation, but a weak inverse correlation with temperature and pressure. FR does not have strong correlation with the pressure and temperature but has a weak correlation with the bico basis weight and % PP. AP has a strong inverse correlation with the bico basis weight and does not have significant correlation with the other factors. Tenacity has a positive correlation with the temperature, indicating that tenacity increased with the raise in temperature, but it has a negative correlation with the bico basis weight. The tenacity values decreased with the increase of bico basis weight, as the MB web would not be expected to contribute notably to the strength of the laminate.

Contour plots of the predicted values for SM laminates:

FR values reach a maximum at higher bico basis weights for SM laminates. Lower basis weights of the bico web and lower % PP present in the bico PP/PE MB web result in lower FR values and higher bico basis weights and a higher % PP result in higher FR. The FR reaches a minimum at 100 % PP and 20-gsm bico basis weight.

Increase in the bico basis weight while maintaining the % PP at a constant level increases the values of FR as shown in Figure 19. At lower % PP in bico MB web; bico basis weight has a pronounced effect on FR of SM laminates than that of % PP and at higher PP percentages the effect of % PP is more pronounced than that of bico basis weight. Observing the contour plot of HH with bico basis weight and % PP in Figure 20 shows that HH increases as the basis weight of the bico web increases. Increase in % PP does not have significant effect but higher HH values were observed at 67.5 % PP. The increase in HH values may be due to the hydrophobic nature of the PP. Higher basis weight of bico results in higher HH values as the increase in weight per unit area may enhance the HH values. The contour plot in Figure 21 shows that AP values increase with a decrease in bico basis weight and the AP values are minimum at 80% PP when basis weight of the bico MB web is maintained at a constant level. A decrease in the values of AP was observed with an increase in bico basis weight when the % PP was maintained at a constant level. The contour plot in Figure 22 shows that the tenacity values are higher between 65 – 85% PP at lower basis weights and the increase in bico basis weight does not have a significant effect. The increase in the % PP results in increase in the tenacity but the effect is comparatively insignificant when compared to other responses such as FR, HH and AP. The contour plot in Figure 23 shows that the FR values were higher at 75% PP and at a bonding temperature of 275 °F. The FR values increased as the % PP increased from 25 to 75% over the temperature range of 250° F – 275° F and decreased over the temperature range of 275° F – 300° F. From Figure 24, it can be

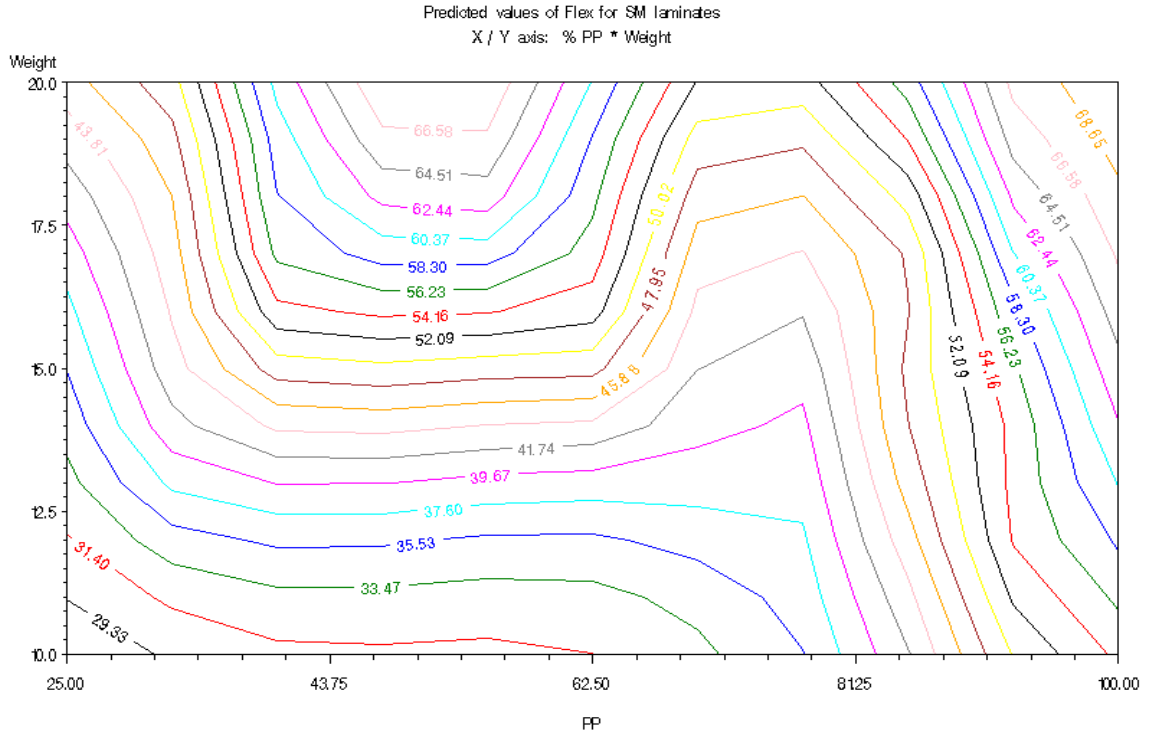


Figure 19: Effect of % PP and bico MB basis weight on flexural rigidity

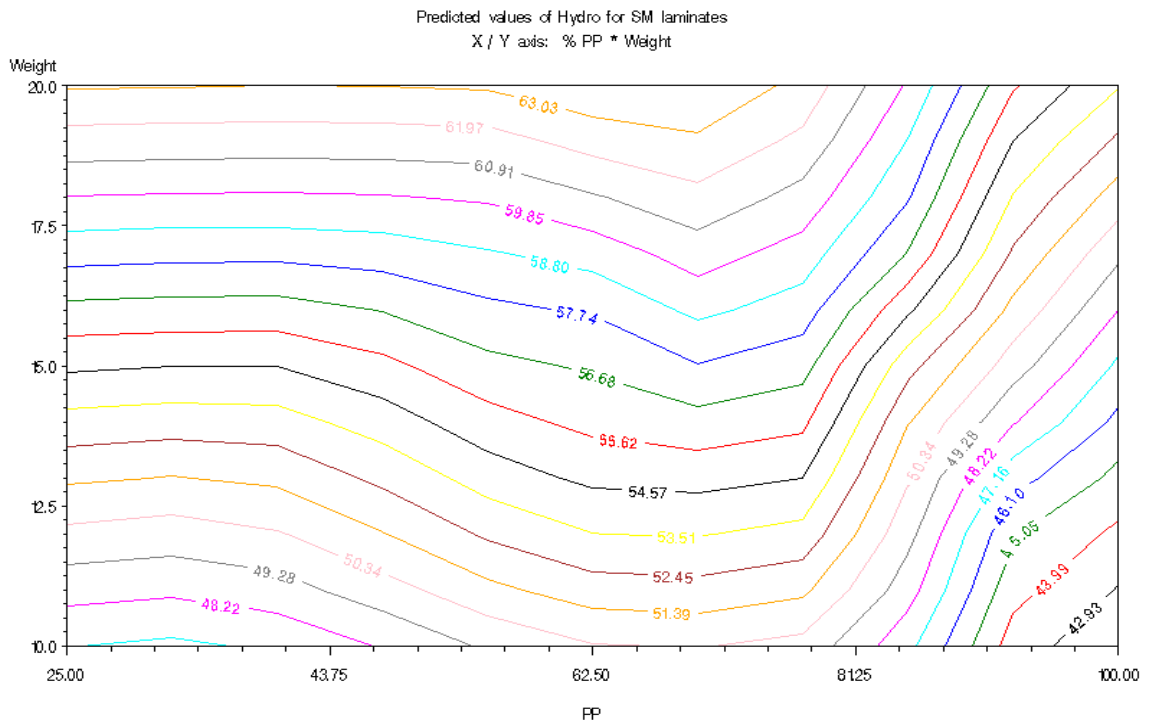


Figure 20: Effect of % PP and bico MB basis weight on hydrostatic head

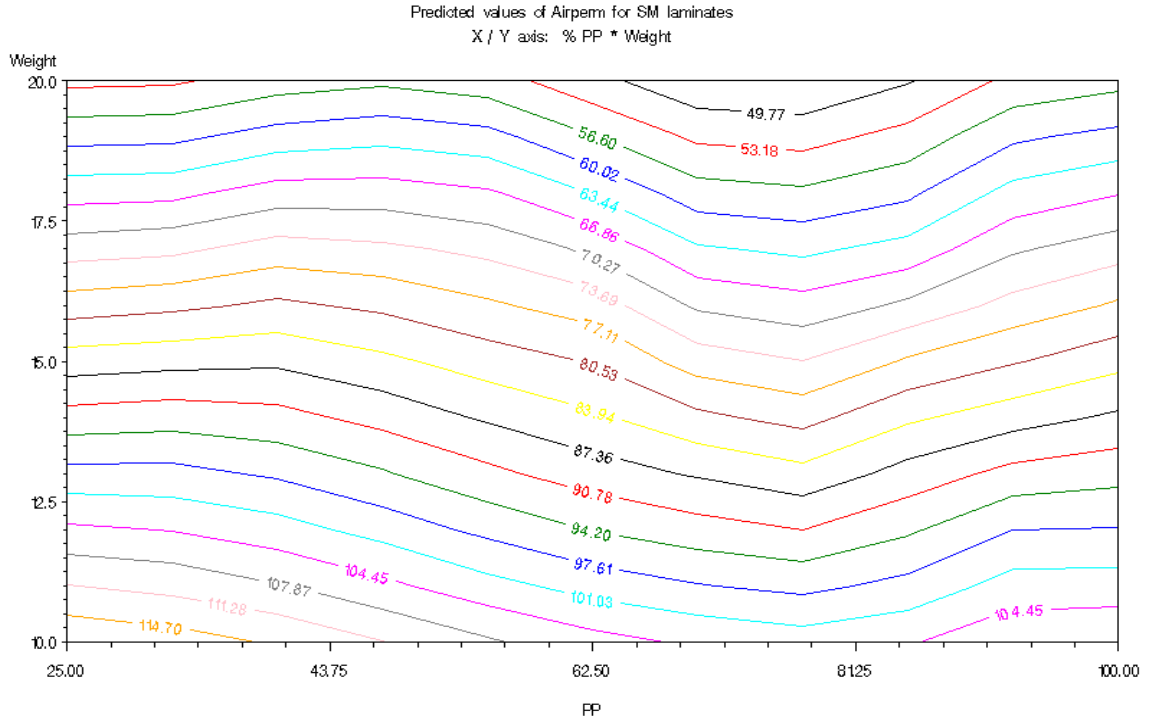


Figure 21: Effect of % PP and bico MB basis weight on air permeability

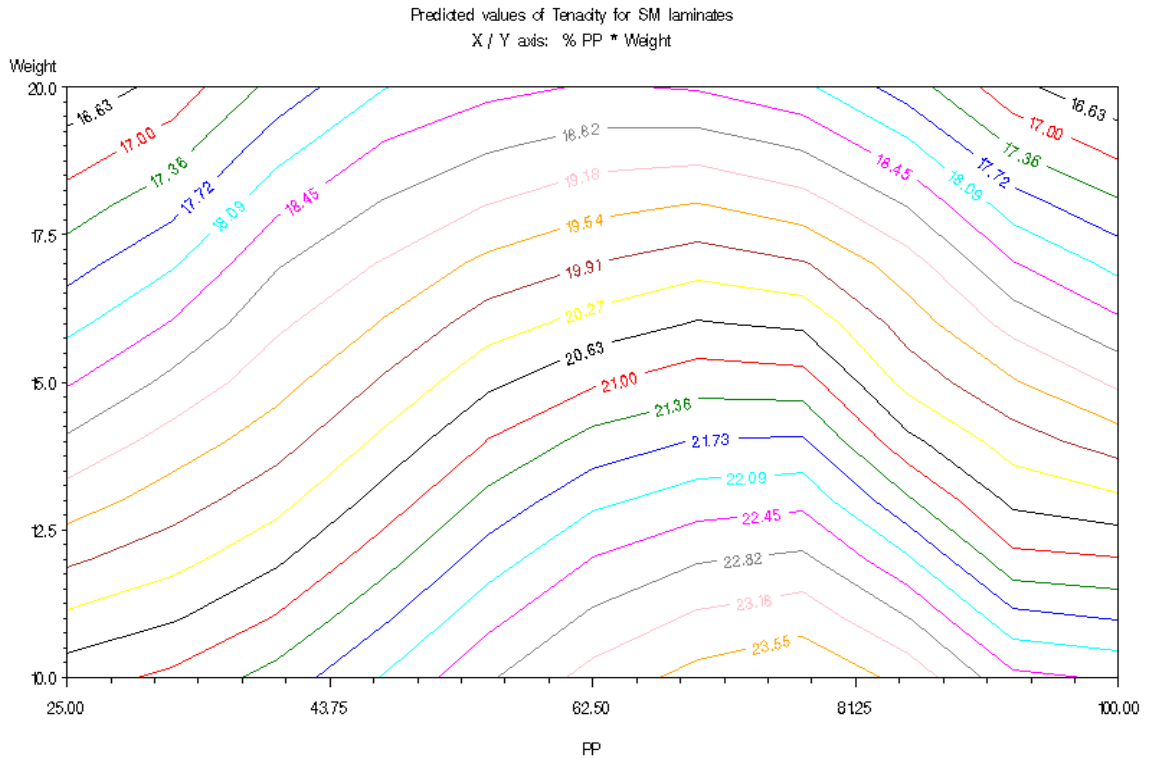


Figure 22: Effect of bico basis weight and % PP on tenacity

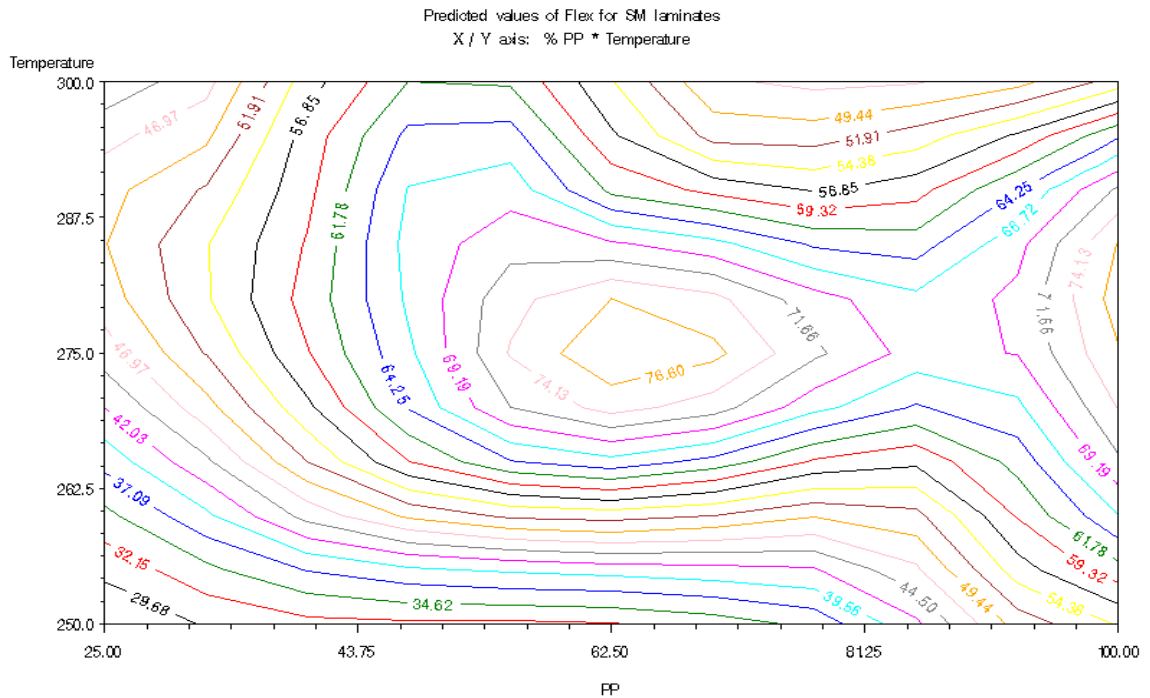


Figure 23: Effect of % PP and temperature on flexural rigidity

observed that the webs containing lower %PP bico MB have lower AP values at higher temperatures and the variation in the AP values was not significant. Higher AP values were obtained at 65% PP. The effects of increasing the temperature from 275° F to 300° F was not significant as can be observed from Figure 24. Lower HH values of SM laminates were predicted at lower and higher PP percentages of MB web and at higher temperatures but higher HH values were observed at 75% PP and at moderate or medium temperature of 275 °F as can be seen in Figure 25. It can be clearly observed from the contour plot in Figure 26 that tenacity values are minimum at 65–75% PP and at a bonding temperature of 275 °F. The variation in tenacity values is not significant from an engineering point of view.

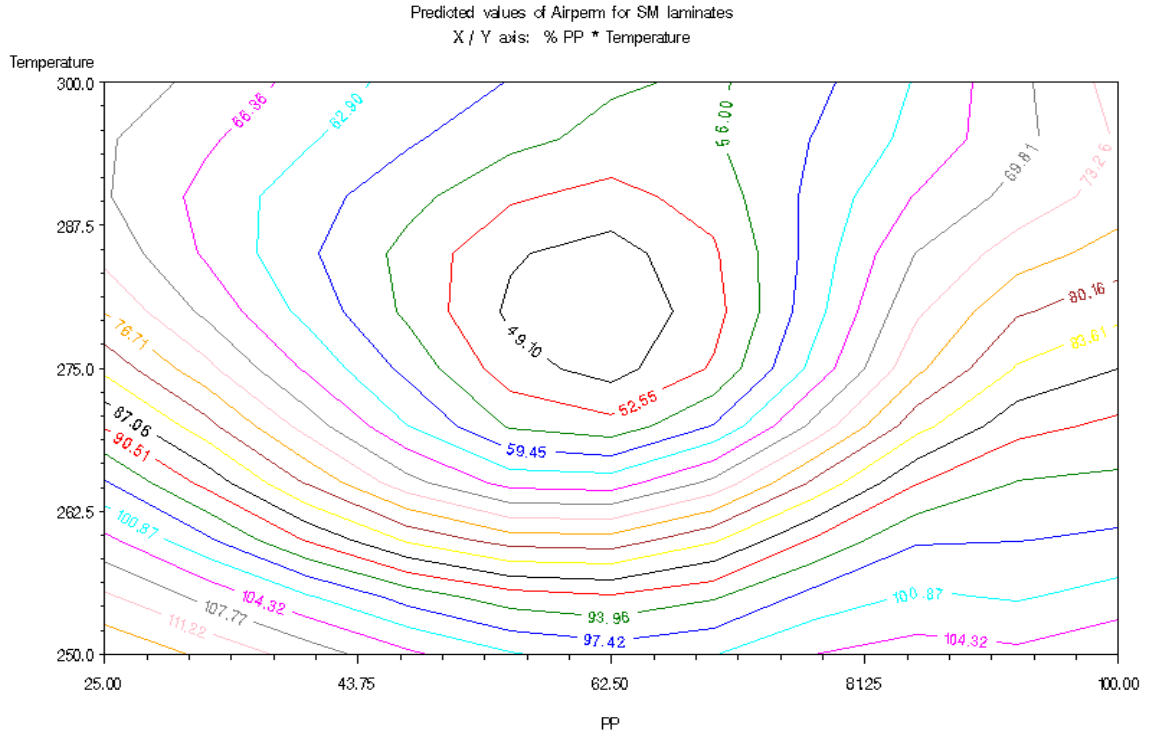


Figure 24: Effect of temperature and % PP on air permeability

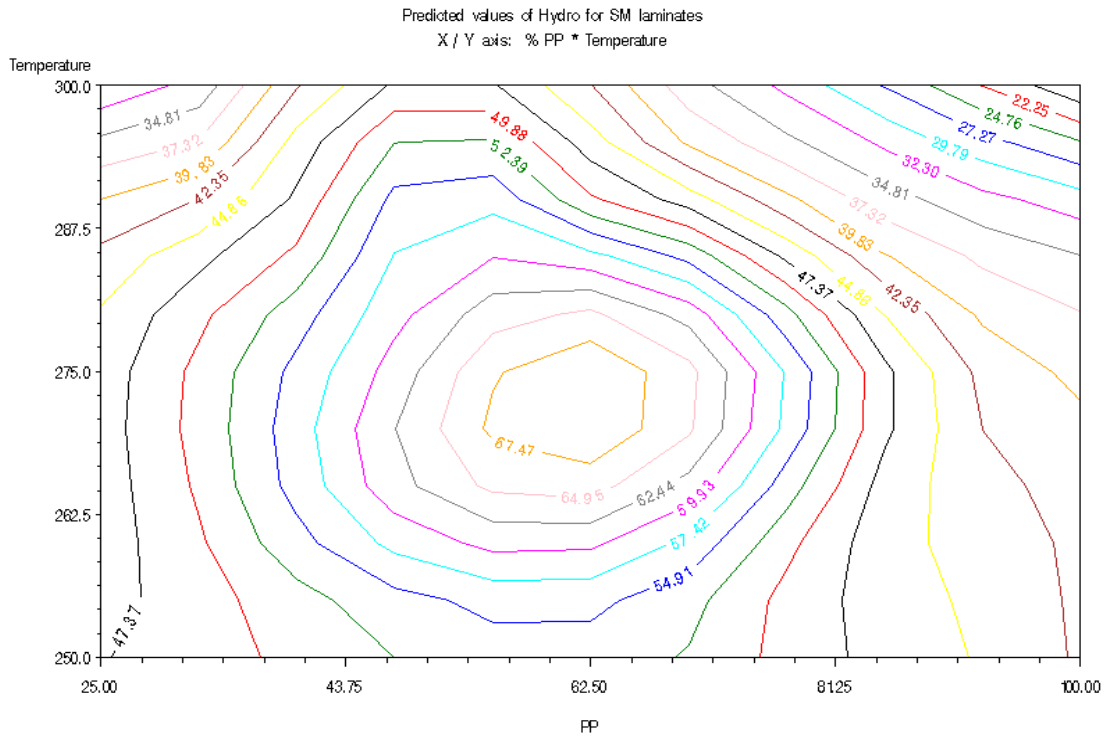


Figure 25: Effect of temperature and % PP on hydrostatic head

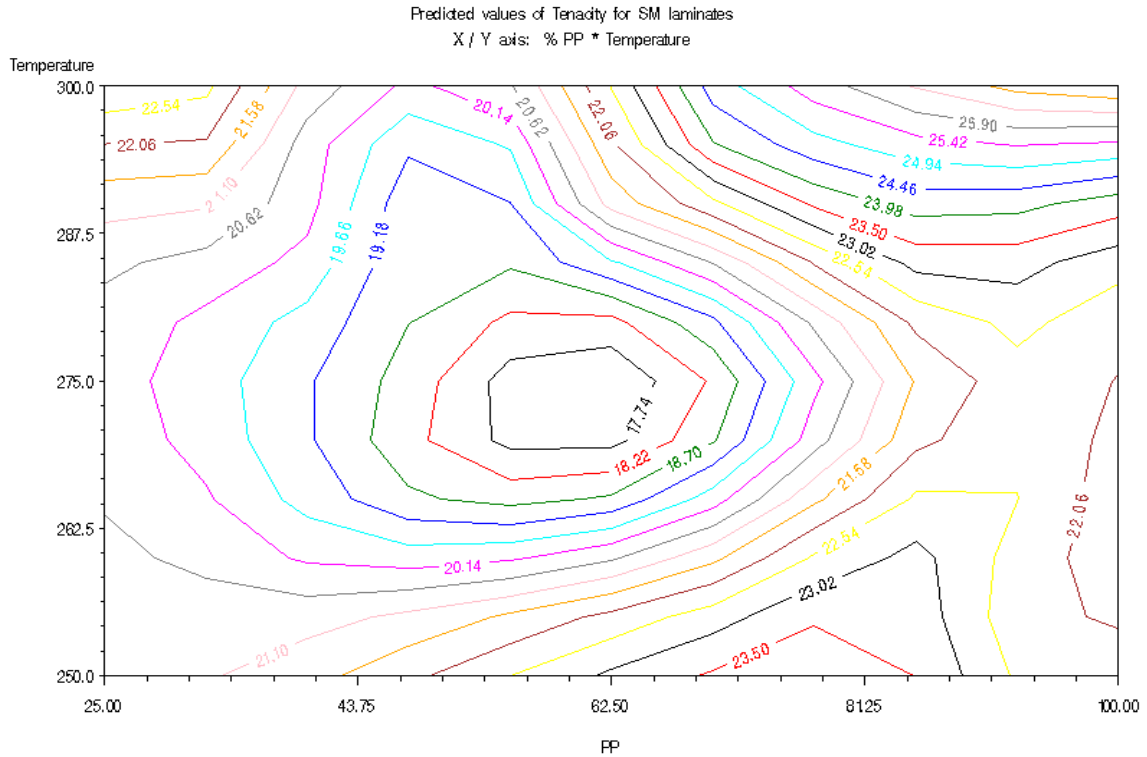


Figure26: Effect of % PP and temperature on tenacity

Contour plots of the predicted values for SMS laminates:

As can be seen in Figure 27 higher basis weights of the bico MB webs and higher %PP resulted in high FR values for SMS laminates. Higher % PP does not have significant effect on FR values of SM laminates. The increase in FR values for SMS laminates may be due to the compact adhesion/bonding of the bico MB web to the laminate. From Figure 28, the maximum HH is observed at high bico basis weights and when the content of PP in the bico MB web is 65%. As the % PP in the bico MB web is increased from 65 to 100, HH values dropped significantly. Similar effect was observed for SM laminates. It may be due to the higher brittle structure developed in the laminate because of the higher % PP in the bico MB web. HH values did not vary significantly at lower basis weights and at lower % PP values. From Figure 29, higher

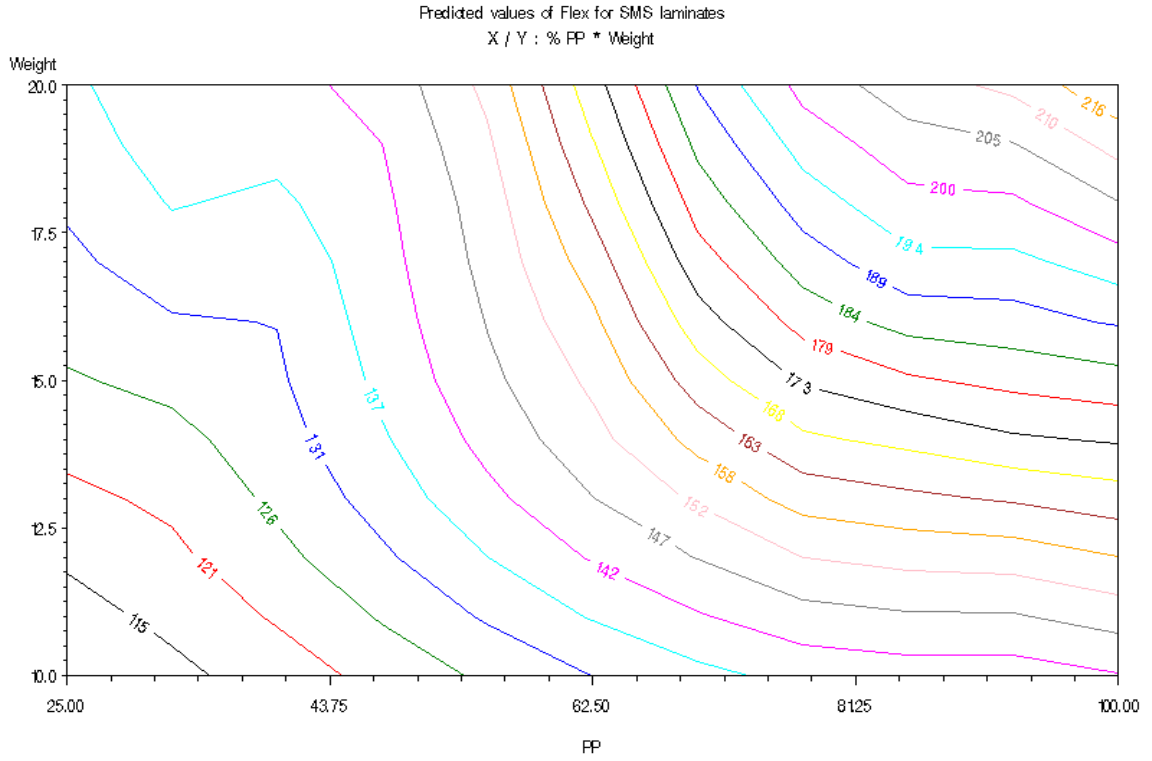


Figure 27: Effect of %PP and bico basis weight on flexural rigidity

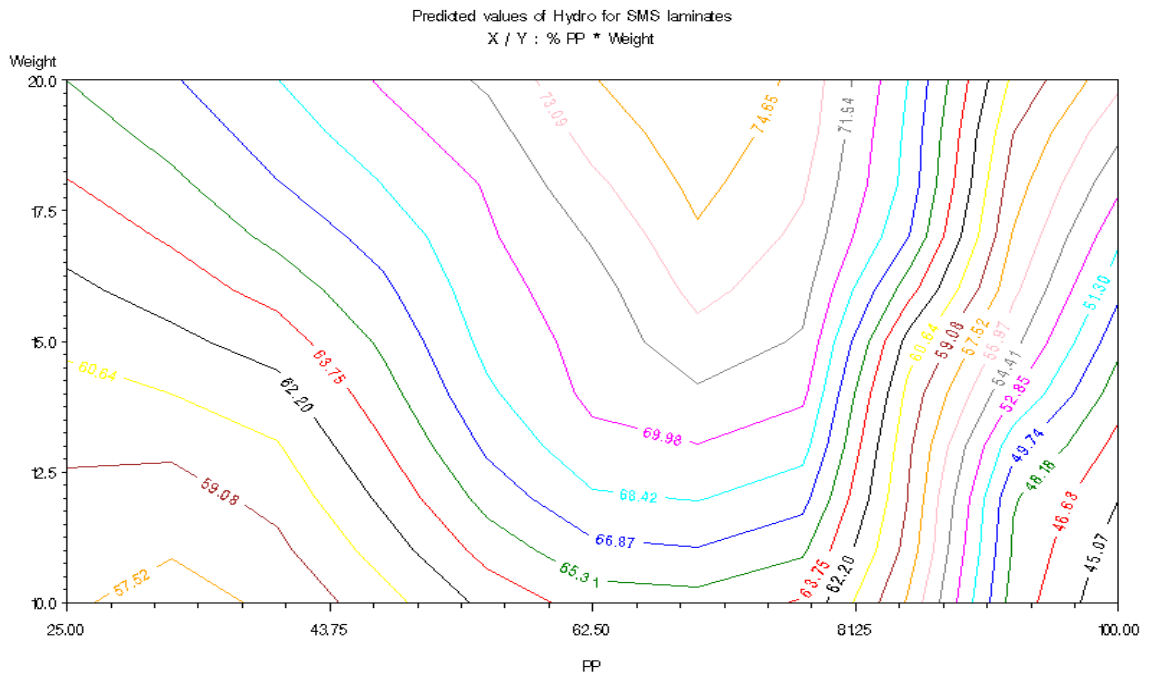


Figure 28: Effect of %PP and bico basis weight hydrostatic head

AP values were observed at lower bico basis weights and the effect of % PP on AP was notably small. Similar effect was observed for SM laminates indicating that higher bico basis weights result in lower AP values. From Figure 30, a higher % PP in the bico MB web and higher bico basis weights resulted in higher tenacity values. An inverse effect to that of SM laminates was observed as the SB web plays a major role in the tensile strength of the laminate. From Figure 31, lower FR values were observed at higher temperatures. Maximum FR was observed at 275 °F and when the PP content in the bico web was 65 %. A similar trend was observed for SM laminates but the FR values of SMS laminates was double to that of SM laminates. From Figure 32, Higher HH values were observed at medium temperatures and at a level of 65% PP in the bico MB web. From Figure 33, it can be seen that no significant variation of AP with the temperature was observed, as was in the case of SM laminates. Minimum AP was observed at 65% PP content in the bico MB web in the SMS laminate. AP values decreased on either side of the local maximum. Higher temperatures and higher % PP content in the MB web helped in higher tenacity values of the SMS laminates as can be observed from Figure 34. Similar effects were observed for SM laminates. Average tenacity values of the SMS laminates were 1.5 times greater than that of SM laminates.

Optimization of the laminating conditions:

Although several other tests of the SM and SMS composites were performed, only air permeability, hydrostatic head, tensile strength and flexural rigidity of the composites were utilized as responses for optimization. After the percentage of PP in bico MB

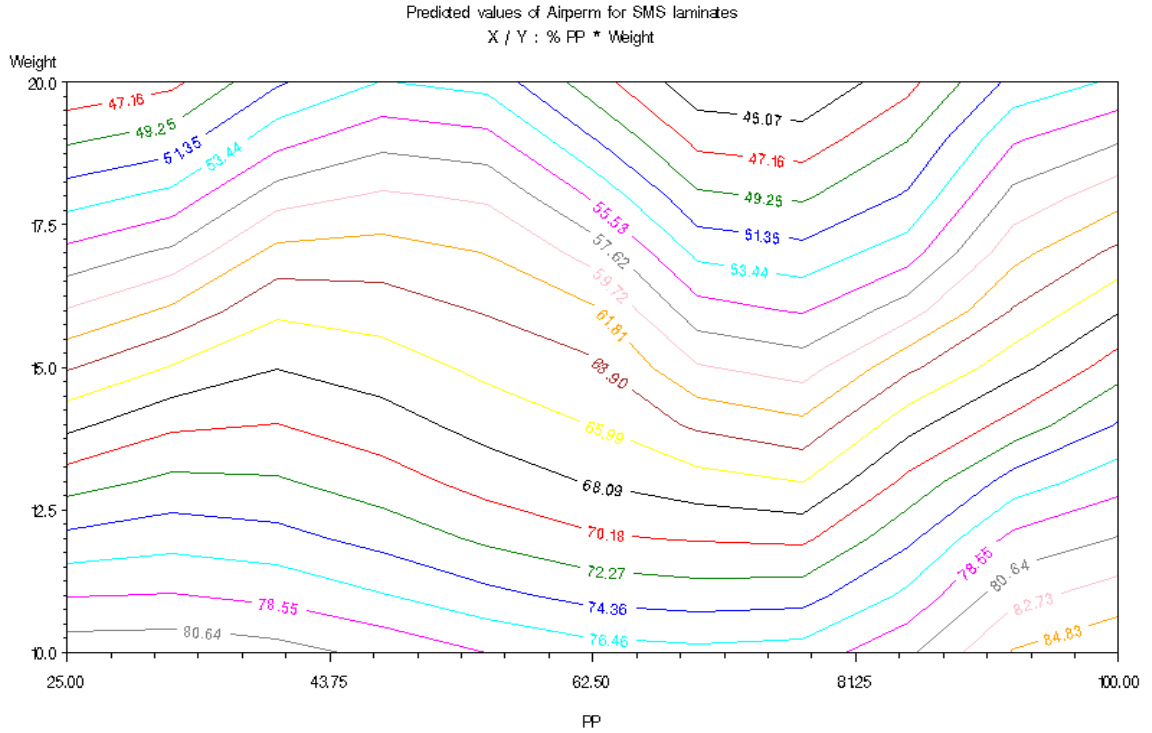


Figure 29: Effect of %PP and weight on air permeability

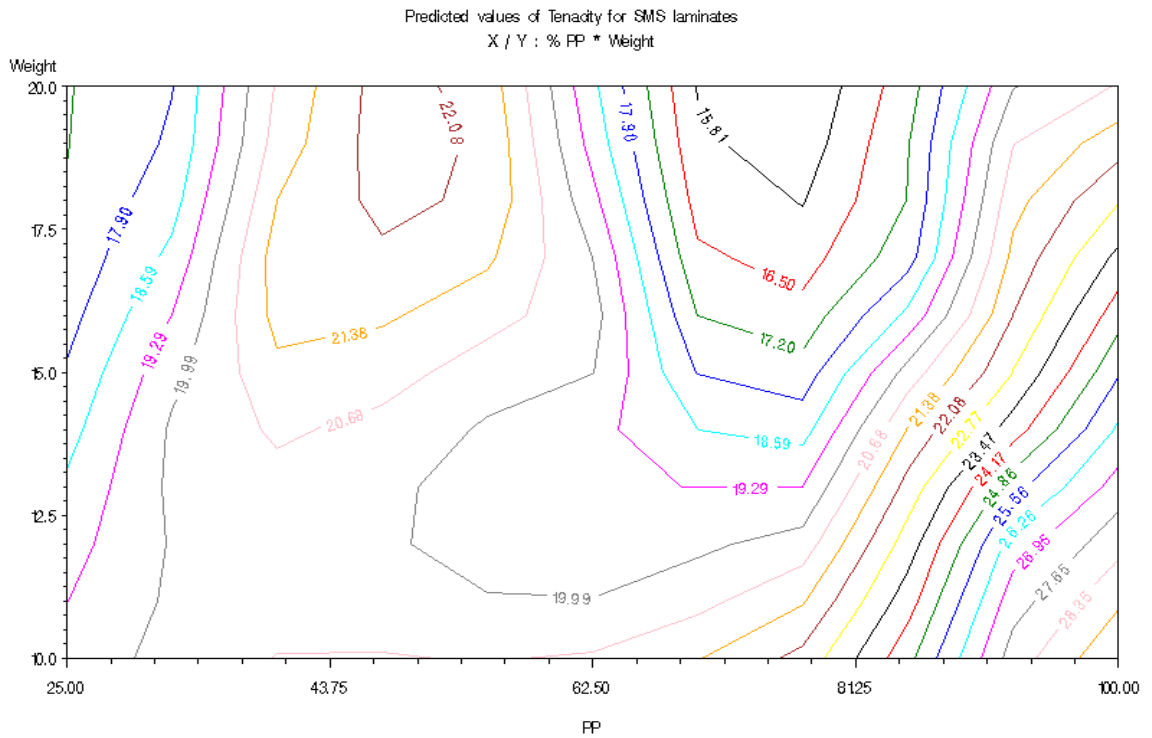


Figure 30: Effect of %PP and bico basis weight on tenacity

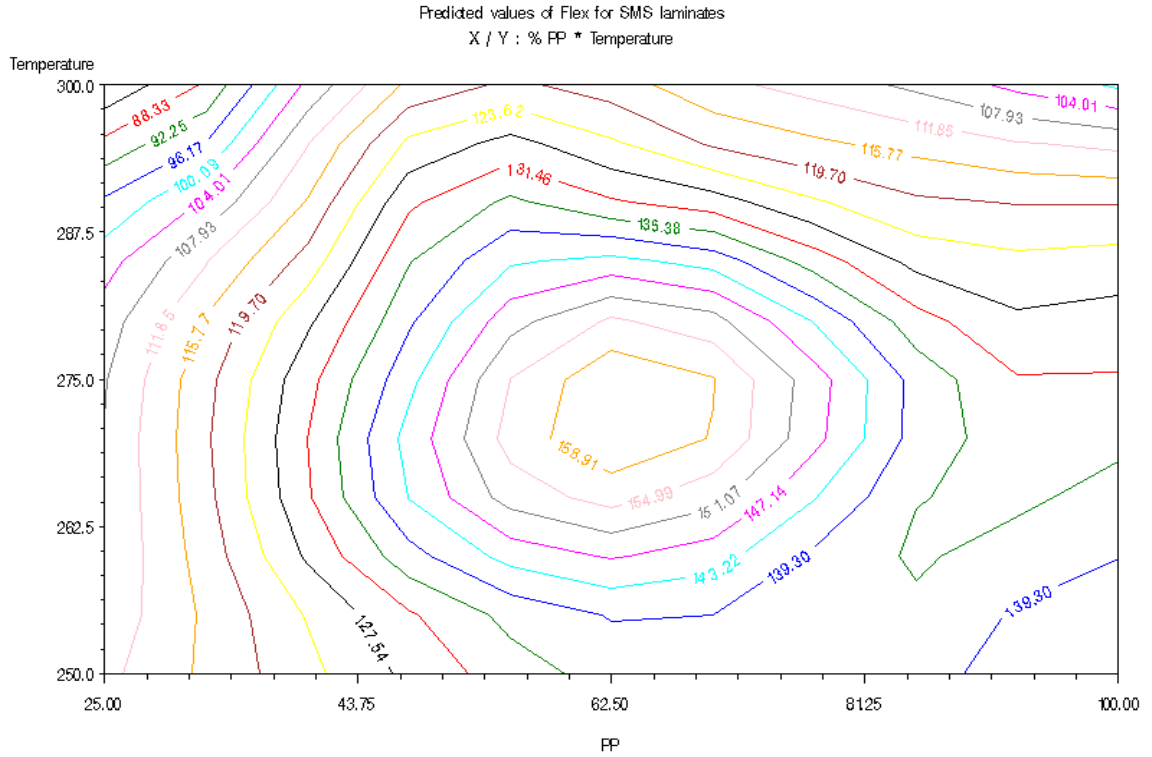


Figure 31: Effect of %PP and temperature on flexural rigidity

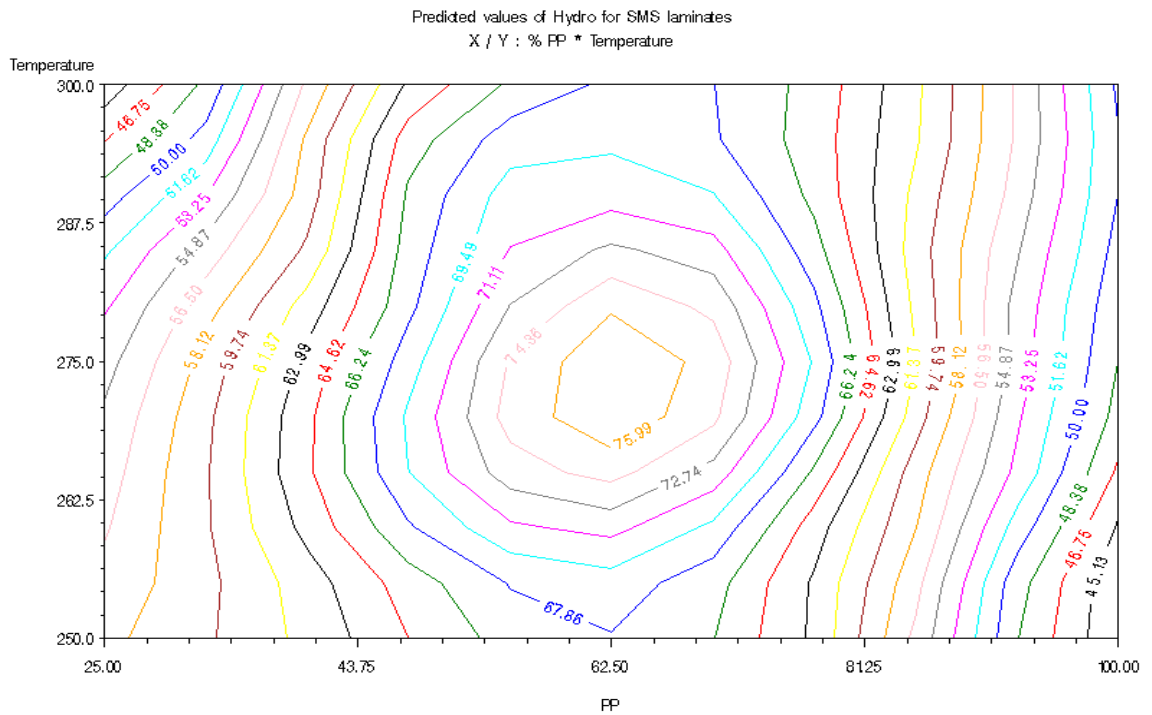


Figure 32: Effect of temperature and %PP on hydrostatic head

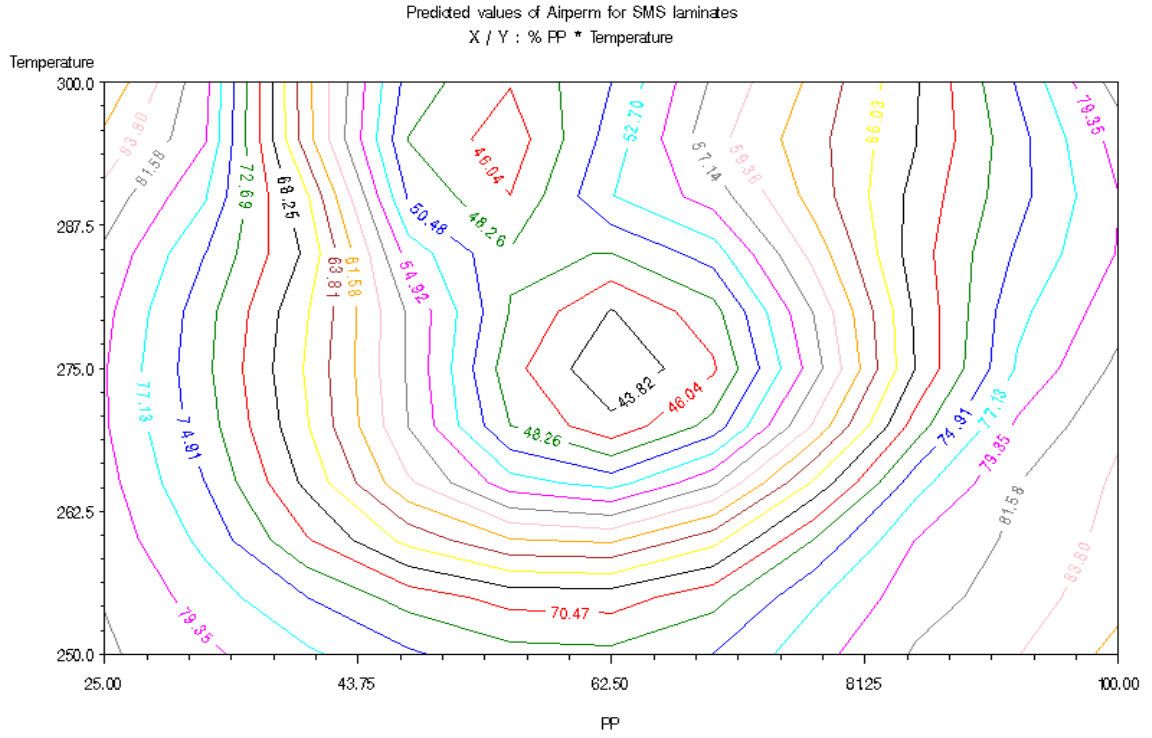


Figure 33: Effect of %PP and temperature on air permeability

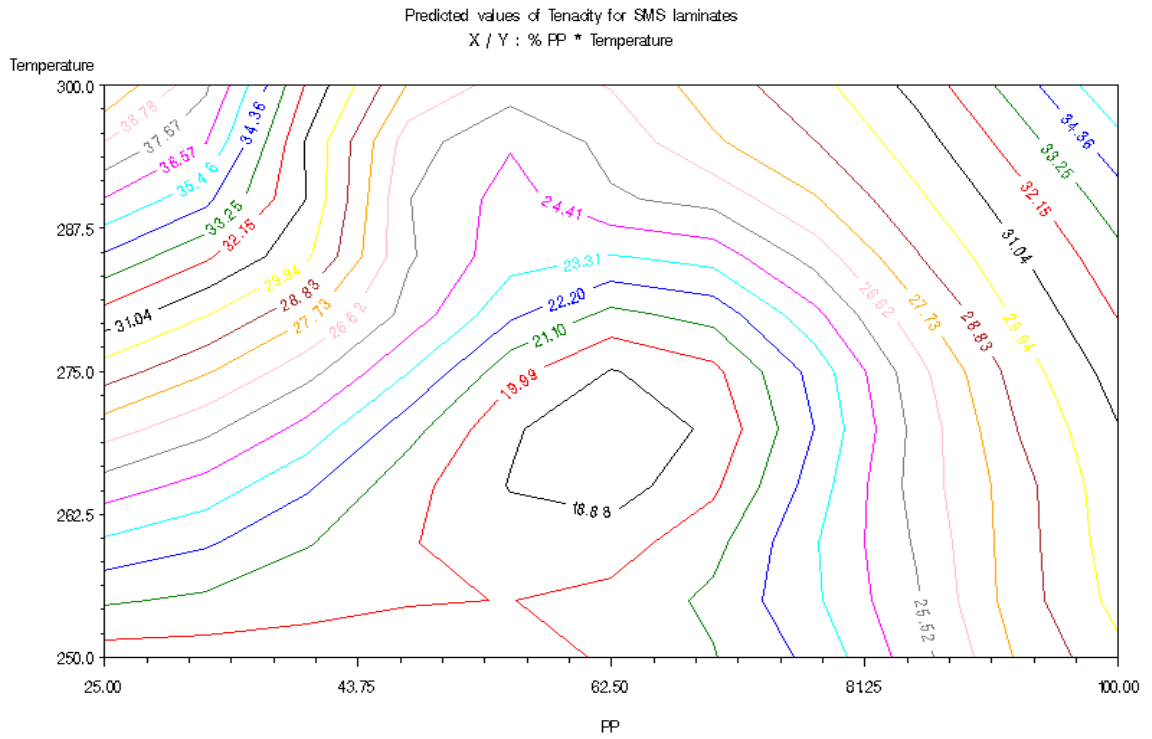


Figure 34: Effect of %PP and temperature on tenacity

web of 50%, bonding temperature is the most significant factor, and HH reached its maximum value at temperatures ranging from 250° F to 280°F. The bonding pressure between 240 PLI and 280 PLI was not significant. If PE was the main component in the MB web, a lower pressure would be required to attain the better end-use properties, i.e. higher air permeability and tenacity, In this case, increasing bonding pressure will result in decrease in AP and tenacity without affecting HH. As the %PP becomes greater than 50%, higher temperature and pressure are needed to achieve the composite with optimum properties. It was obviously because of higher melting temperature of PP. However, a temperature higher than 280° F, will more likely lead to lower HH and to higher AP and tenacity, which may be due to the pinhole formation at the edge areas of bonding area. Prediction profile graphs were drawn using JMP software. The optimum processing conditions of bonding temperature, calender nip pressure, % PP and bicomponent basis weight for optimum properties were shown in the Figures 35 – 39. Figure 35 shows the prediction profile for FR revealing that temperature and pressure do not have significant effect on FR. On the other hand % PP and bico basis weight have a significant effect on FR. FR is lower at lower % PP and at lower bico basis weights. Figure 36 show that the bonding pressure and %PP do not have significant effect on the AP of the laminate. The effect of temperature and bico basis weight can be seen from the above profiler graphs drawn using response surface modeling. All four factors have a significant effect on the HH of the laminates. Temperature, % PP and bico basis weight have a significant effect; whereas bonding pressure has a marginal effect on the HH of the laminates, as can be seen in Figure 37. Figure 38 shows that tenacity as affected by both bonding

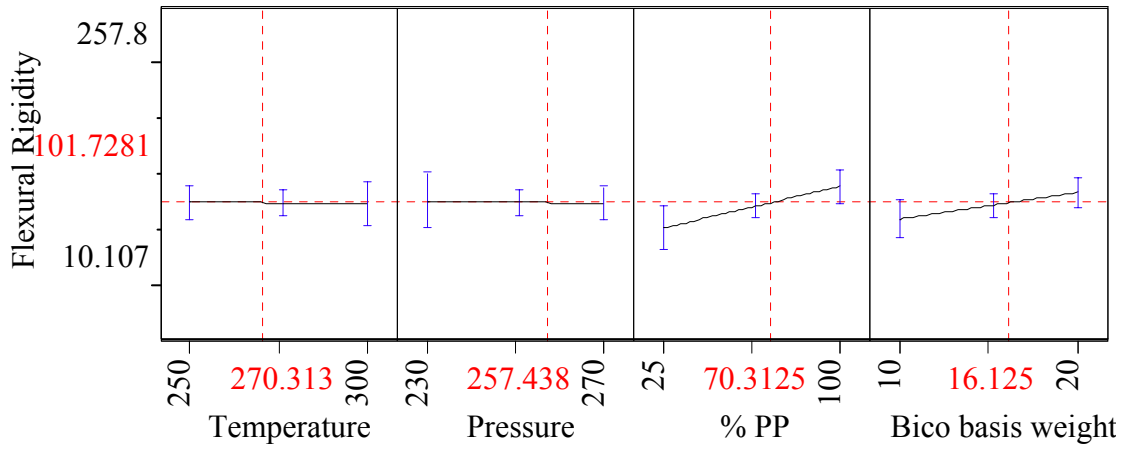


Figure 35: Prediction profile for flexural rigidity as affected by bonding temperature, calender nip pressure, % PP and bico MB basis weight

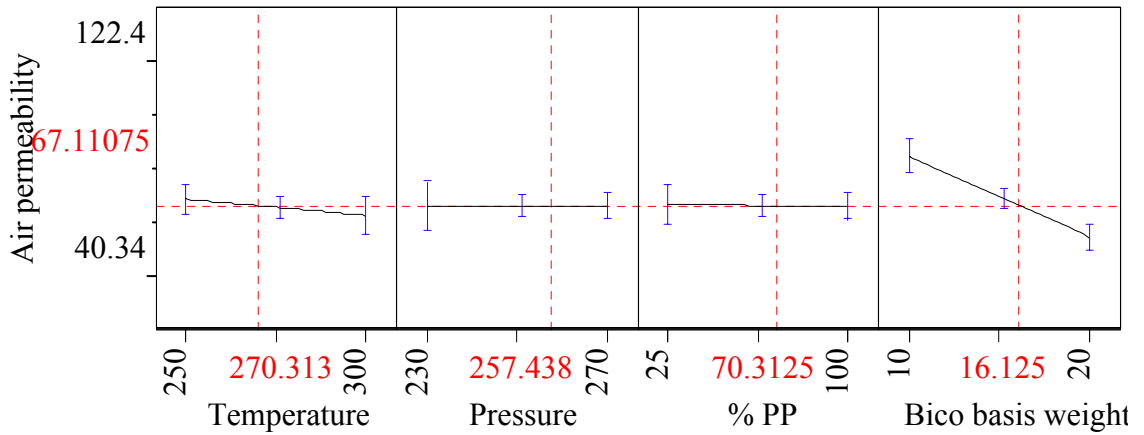


Figure 36: Prediction profile for air permeability as affected by bonding temperature, calender nip pressure, % PP and bico MB basis weight

temperature and bico basis weight whereas % PP and calender nip pressure has a minimum effect on the tenacity and strength of the laminates. The prediction profile graphs with varying proportions of % PP for FR are shown in Figures 39 - 45. The prediction profile graphs for HH with varying proportions of % PP are shown in Figures 46–53.

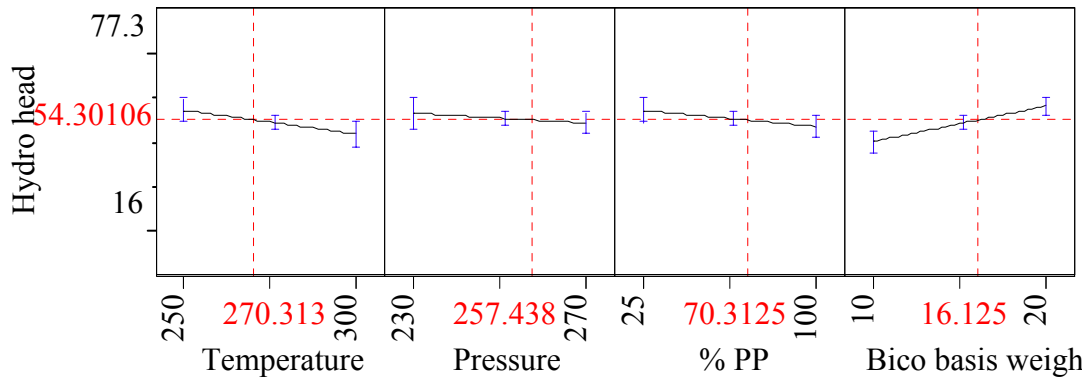


Figure 37: Prediction profile for HH as affected by bonding temperature, calender nip pressure, % PP and bico MB basis weight

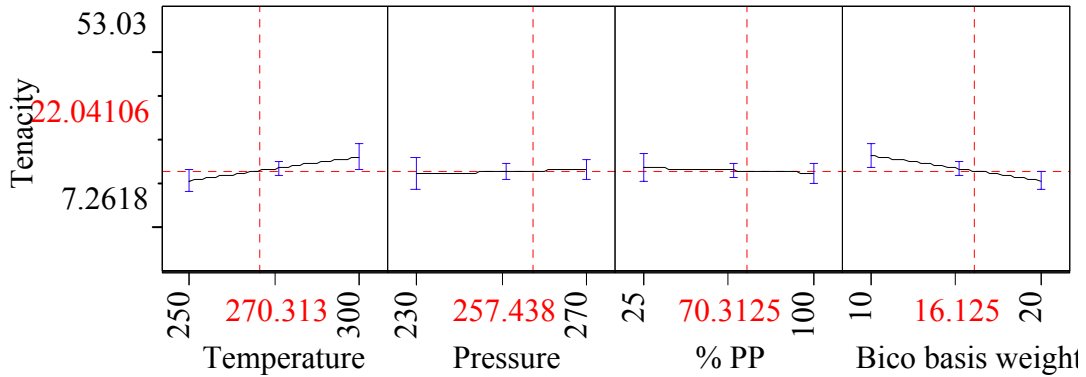


Figure 38: Prediction profile for tenacity as affected by bonding temperature, calender nip pressure, % PP and bico MB basis weight

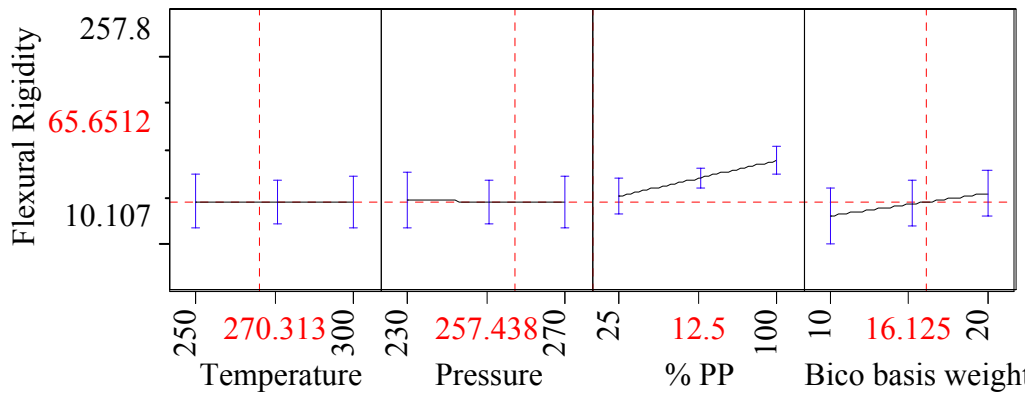


Figure 39: Prediction profile of FR with 12.5% PP

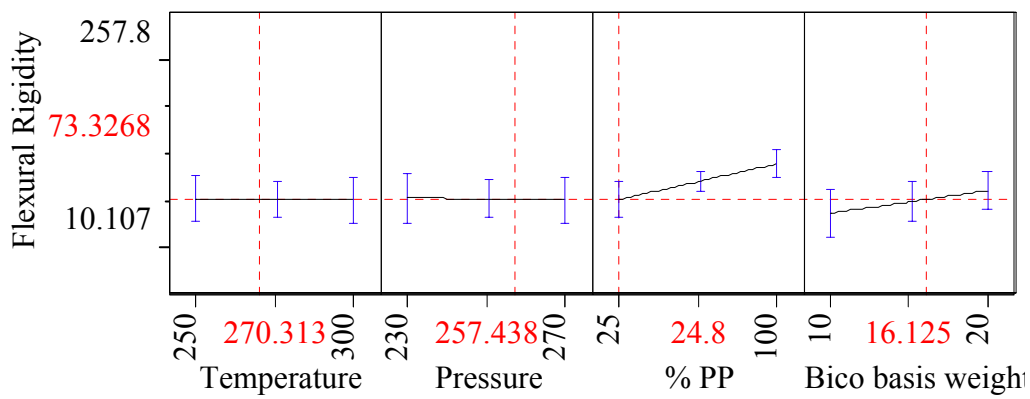


Figure 40: Prediction profile of FR with 24.8% PP

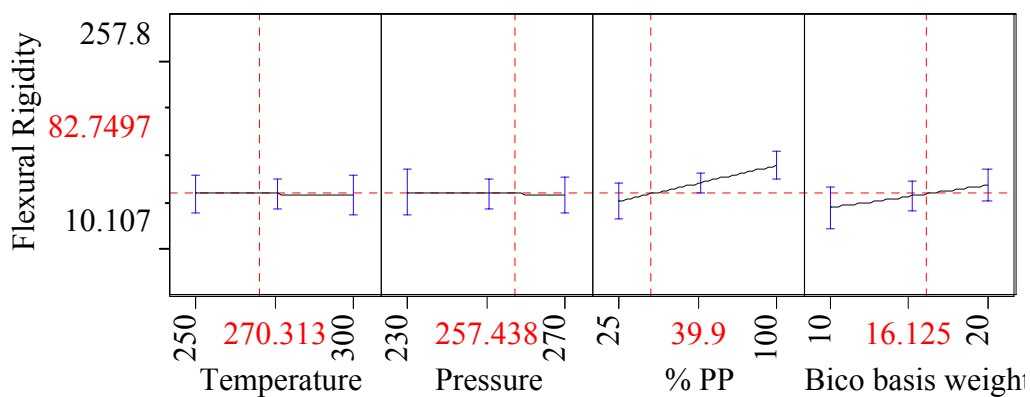


Figure 41: Prediction profile of FR with 39.9% PP

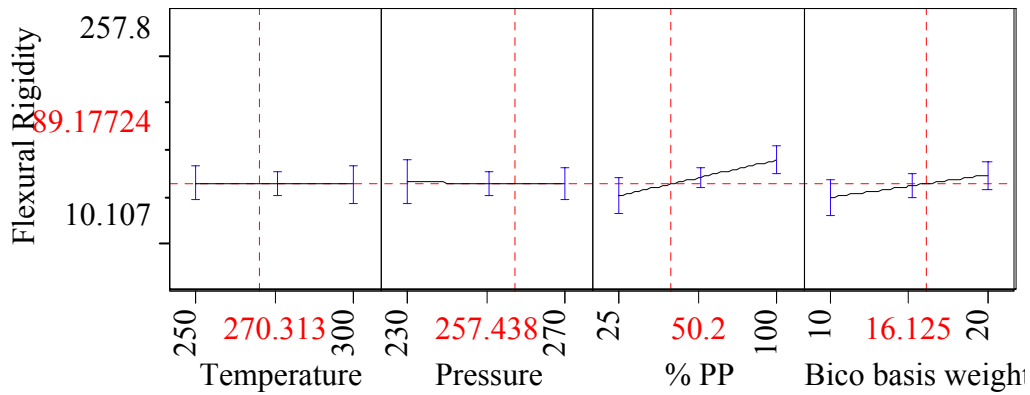


Figure 42: Prediction profile of FR with 50.2% PP

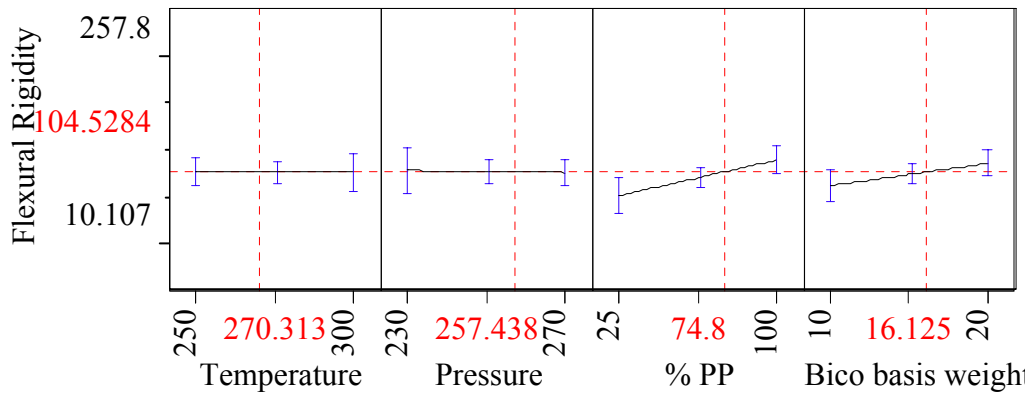


Figure 43: Prediction profile of FR with 74.8 %PP

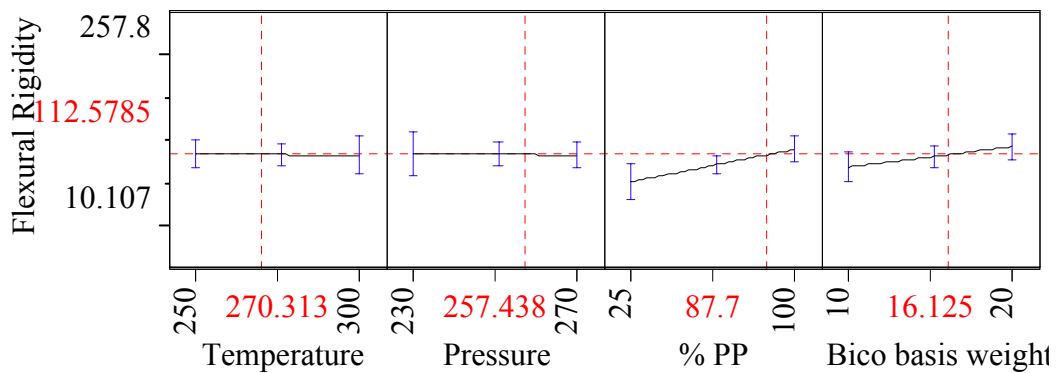


Figure 44: Prediction profile of FR with 87.7% PP

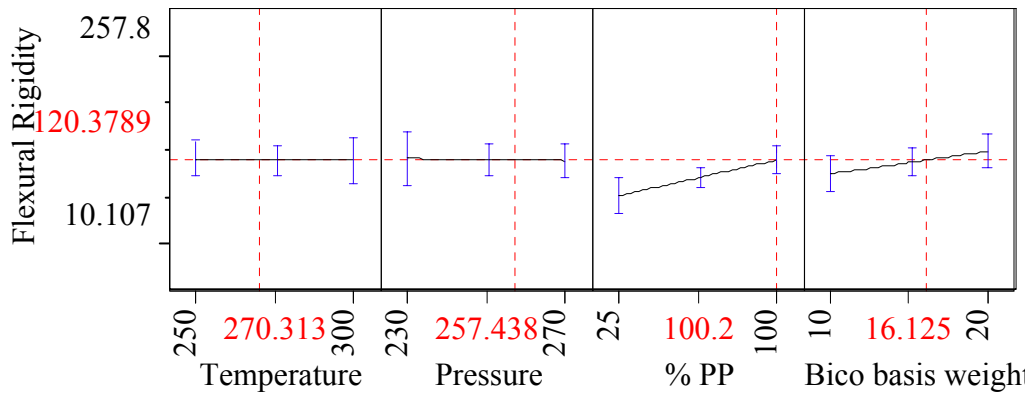


Figure 45: Prediction profile of FR with 100% PP

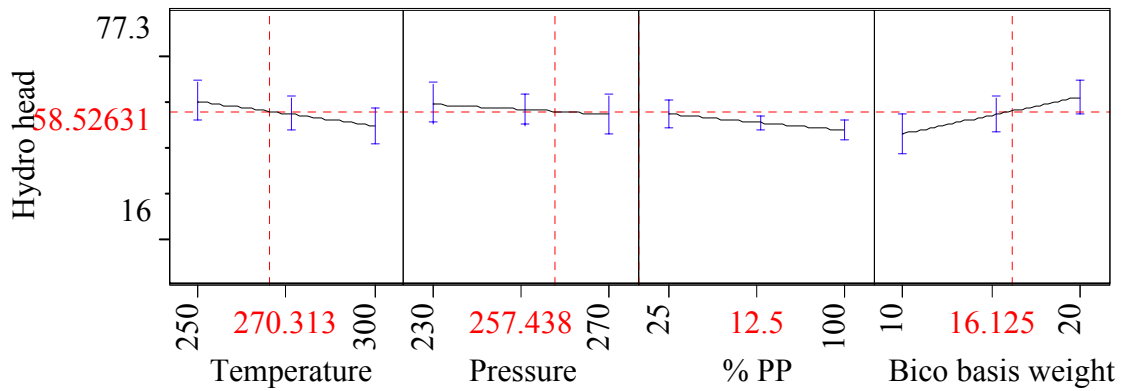


Figure 46: Prediction profile of HH with 12.5% PP

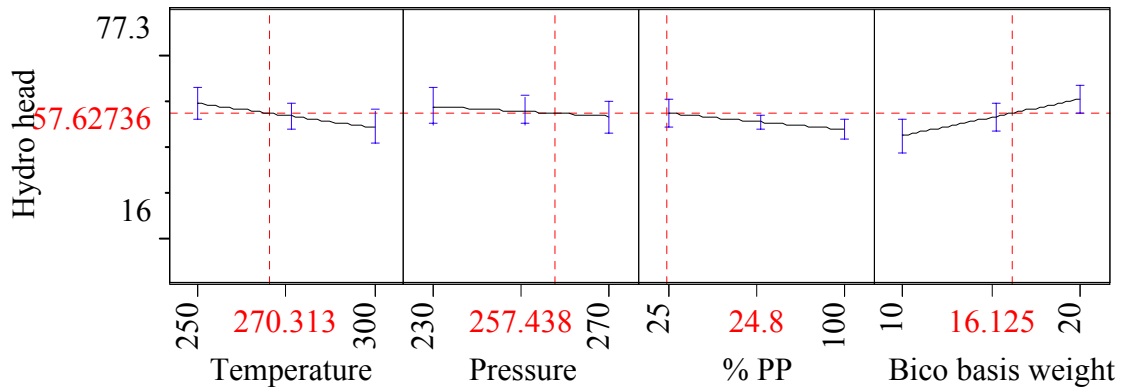


Figure 47: Prediction profile of HH with 24.8% PP

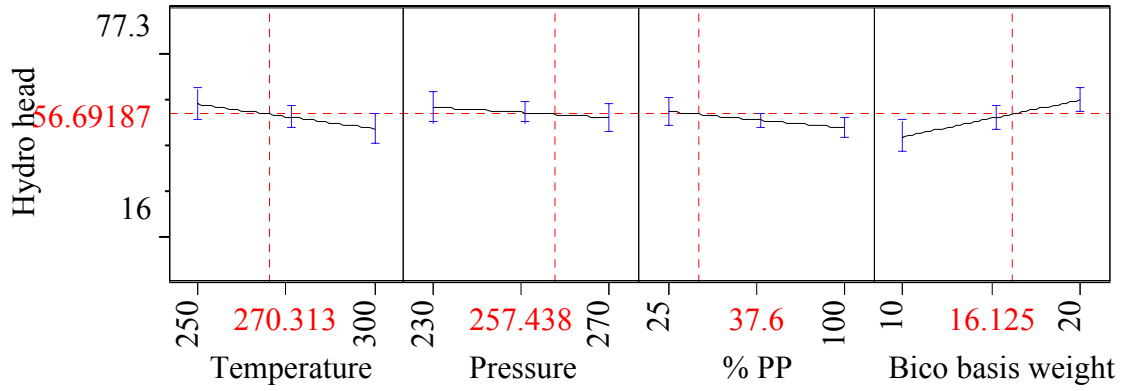


Figure 48: Prediction profile of HH with 37.6% PP

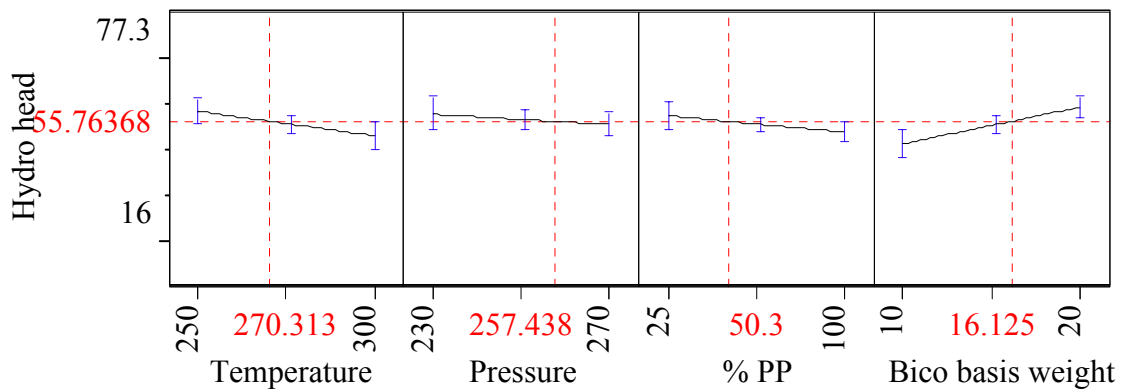


Figure 49: Prediction profile of HH with 50.3% PP

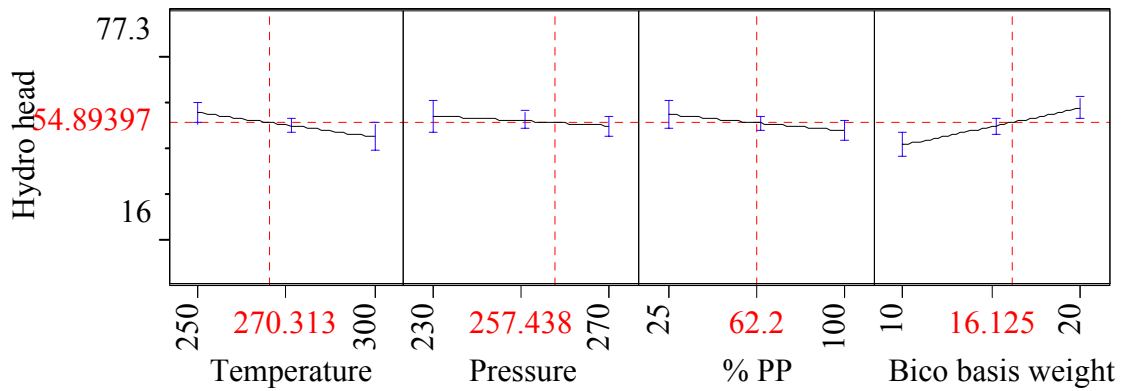


Figure 50: Prediction profile of HH with 62.2% PP

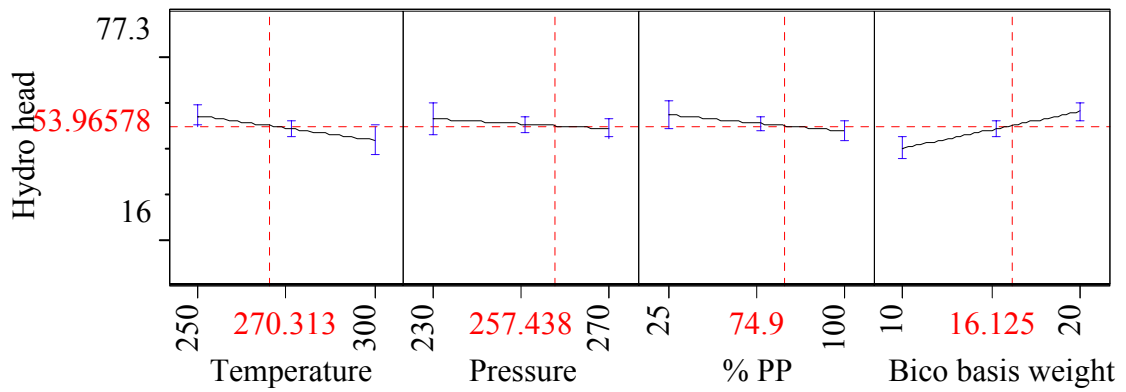


Figure 51: Prediction profile of HH with 74.9% PP

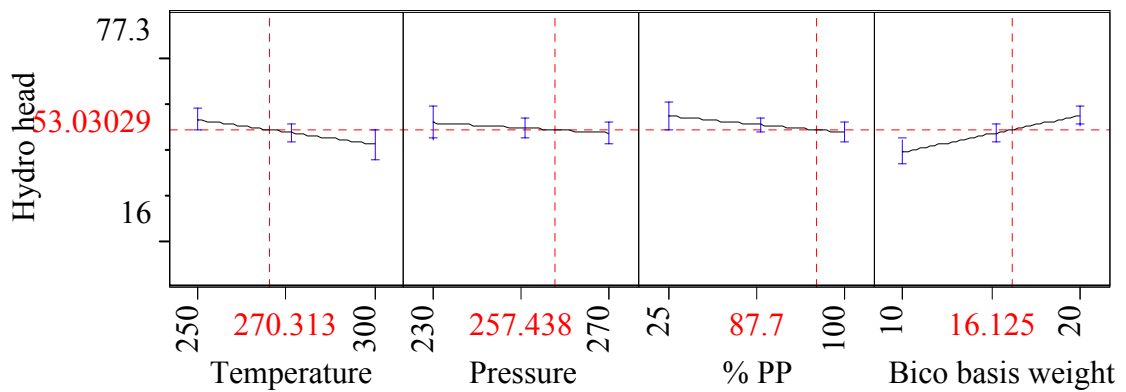


Figure 52: Prediction profile of HH with 87.7% PP

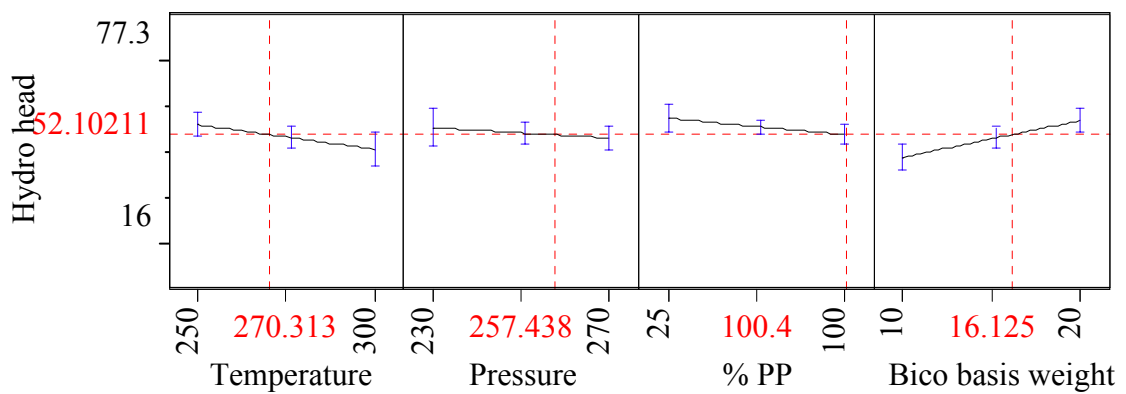


Figure 53: Prediction profile of HH with 100% PP

CHAPTER V

SUMMARY & CONCLUSIONS

As the % of PP in the web was increased, better mechanical properties were observed and the optimum percentage of PP is 70 – 75 % of the bico MB web and the increase in percentage of the web thereafter does not have notable effect. The effect of %PP in the bico MB web was more pronounced in SM laminates than in SMS laminates. This is probably due to the much greater strength of SB than MB in SMS webs.

Possible optimal production conditions were suggested. As the % of PP in bico PP/PE MB web increased, the best barrier properties were achieved at increased bonding temperature and pressure in the production of SM composites with the MB component on the bottom side against the smooth roll. When the SM was produced with the MB on the top of SB so that the MB directly contacts the heated metal roller, over-bonding may occur at relatively higher bonding temperature. This, however, may be avoided by increasing the production speed. The Response Surface Design method was successfully used in this research, which provides a feasible way in optimizing a process involving multiple factors and is an efficient way of narrowing down the optimum properties for the required end uses applications. The production of bico PP/PE MB web containing SMS composites is not sensitive to the bonding conditions in the experimental ranges studied. However, because of the lower melting point of PE, it is expected that the advantage of bico PP/PE MB would be more notable with higher basis weight. Thus, it would be feasible to produce SMS with a higher weight of MB and with better barrier properties without decreasing the SMS production

speed. SM and SMS laminates containing 100% PP were produced. The effect of temperature and calender nip pressure does not have notable effect on the properties of the webs containing 100%PP. The variation in the values of the properties between SM and SMS is chiefly due to the variation due to the number of layers present in them. HH and AP values were significantly affected by the increase in % PP and bico basis weight. The type of the laminate has a significant effect on all the properties. Increase in bico basis weight has resulted in increase of HH, AP and FR. Bonding temperature effects, although not significant, resulted in increases in tenacity and FR values. Bonding pressure did not have any significant effect over the experimental range studied, but it was observed that a higher a pressure yields better mechanical properties. Similar trends of the contour lines for the predicted values were observed for SM and SMS laminates. Higher temperatures and a higher % PP content in the laminates resulted in higher tenacity for both the SM and SMS laminates. Lower FR, lower HH values, higher AP and lower tenacity values were observed when the PP content in bico PP/PE MB web is 65 –70% for SMS laminates. For SM laminates higher tenacity values were observed with 65-70% PP in the bico MB web.

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APPENDIX

Following procedural statements were used for the analysis in SAS

```
proc glm;
```

```
class temp press pp bico type;
```

```
model flex hydro airperm tenacity= temp press pp bico type;
```

```
means temp press pp bico type;
```

```
means temp press pp bico type /tukey;
```

```
run; .
```

```
proc glm;
```

```
class temp press pp bico type;
```

```
model flex = temp press pp bico type pp*bico pp*type bico*type;
```

```
means temp press pp bico type;
```

```
means temp press pp bico type /tukey;
```

```
run; .
```

```
proc glm;
```

```
class temp press pp bico type;
```

```
model hydro = temp press pp bico type temp*pp temp*bico temp*type  
pp*bico pp*type bico*type ;
```

```
means temp press pp bico type;
```

```
means temp press pp bico type /tukey;
```

```
lsmeans pp*bico / /*pdiff*/ slice=bico;
```

```
lsmeans pp*type / /*pdiff*/ slice=type;
```

```
run; .
```

```
proc glm;
```

```
class temp press pp bico type;
```

```
model airperm = temp press pp bico type bico*type;
```

```

means temp press pp bico type;
means temp press pp bico type /tukey;
run; .

```

```

proc sort;by pp;
proc glm;by pp;
class bico type;
model hydro = bico type ;
means bico type;
means bico type /tukey;
run; .

```

Significant main effects can be observed from the below data:

Dependent Variable: flex

Source	DF	Type I SS	Mean Square	F Value	Pr > F
temp	2	11428.5756	5714.2878	3.70	0.0296
press	2	101.3278	50.6639	0.03	0.9677
pp	3	18982.4652	6327.4884	4.10	0.0097
bico	1	11014.4850	11014.4850	7.14	0.0094
type	1	146021.8382	146021.8382	94.64	<.0001

Source	DF	Type III SS	Mean Square	F Value	Pr > F
temp	2	11428.5756	5714.2878	3.70	0.0296
press	2	101.3278	50.6639	0.03	0.9677
pp	3	18982.4652	6327.4884	4.10	0.0097
bico	1	11014.4850	11014.4850	7.14	0.0094
type	1	146021.8382	146021.8382	94.64	<.0001

Dependent Variable: hydro

Source	DF	Type I SS	Mean Square	F Value	Pr > F
temp	2	621.051457	310.525728	4.75	0.0117
press	2	220.391653	110.195826	1.68	0.1930
pp	3	4201.472430	1400.490810	21.41	<.0001
bico	1	1481.806062	1481.806062	22.65	<.0001
type	1	1710.862738	1710.862738	26.15	<.0001

Source	DF	Type III SS	Mean Square	F Value	Pr > F
temp	2	1683.311530	841.655765	12.87	<.000
press	2	59.558914	29.779457	0.46	0.6362
pp	3	2416.434152	805.478051	12.31	<.0001
bico	1	1669.011417	1669.011417	25.51	<.0001
type	1	1710.862738	1710.862738	26.15	<.0001

Dependent Variable: air permeability

Source	DF	Type I SS	Mean Square	F Value	Pr > F	
temp		2	621.051457	310.525728	4.75	0.0117
press		2	220.391653	110.195826	1.68	0.1930
pp		3	4201.472430	1400.490810	21.41	<.0001
bico		1	1481.806062	1481.806062	22.65	<.0001
type		1	1710.862738	1710.862738	26.15	<.0001

Source	DF	Type III SS	Mean Square	F Value	Pr > F
temp	2	1683.311530	841.655765	12.87	<.0001
press	2	59.558914	29.779457	0.46	0.6362
pp	3	2416.434152	805.478051	12.31	<.0001
bico	1	1669.011417	1669.011417	25.51	<.0001
type	1	1710.862738	1710.862738	26.15	<.0001

Dependent Variable: tenacity

Source	DF	Type I SS	Mean Square	F Value	Pr > F
temp	2	499.5578780	249.7789390	4.10	0.0207
press	2	18.6094320	9.3047160	0.15	0.8587
pp	3	429.9717462	143.3239154	2.35	0.0796

bico	1	500.1256046	500.1256046	8.21	0.0055
type	1	592.6305167	592.6305167	9.73	0.0026

Source	DF	Type III SS	Mean Square	F Value	Pr > F
temp	2	474.3934594	237.1967297	3.89	0.0249
press	2	12.8349474	6.4174737	0.11	0.9002
pp	3	246.8840829	82.2946943	1.35	0.2651
bico	1	436.7032199	436.7032199	7.17	0.0092
type	1	592.6305167	592.6305167	9.73	0.0026

Significant interaction terms can be observed in below data:

Dependent variable: Flexural rigidity

Source	DF	Type I SS	Mean Square	F Value	Pr > F
temp	2	11428.5756	5714.2878	3.74	0.0292
press	2	101.3278	50.6639	0.03	0.9674
pp	3	18982.4652	6327.4884	4.14	0.0096
bico	1	11014.4850	11014.4850	7.20	0.0093
type	1	146021.8382	146021.8382	95.47	<.0001
pp*bico	2	1745.7434	872.8717	0.57	0.5680
pp*type	3	4912.5133	1637.5044	1.07	0.3679
bico*type	1	3459.1438	3459.1438	2.26	0.1375

Source	DF	Type III SS	Mean Square	F Value	Pr > F
temp	2	708.51743	354.25872	0.23	0.793
press	2	2481.12434	1240.56217	0.81	0.4489
pp	3	22013.42592	7337.80864	4.80	0.0045
bico	1	12343.42589	12343.42589	8.07	0.0060
type	1	99111.07651	99111.07651	64.80	<.0001
pp*bico	2	2892.02333	1446.01166	0.95	0.3939
pp*type	3	5406.51597	1802.17199	1.18	0.3250
bico*type	1	3459.14382	3459.14382	2.26	0.1375

Dependent variable: Hydrostatic head

Source	DF	Type I SS	Mean Square	F Value	Pr > F
temp	2	11428.5756	5714.2878	3.74	0.029
press	2	101.3278	50.6639	0.03	0.9674
pp	3	18982.4652	6327.4884	4.14	0.0096
bico	1	11014.4850	11014.4850	7.20	0.0093
type	1	146021.8382	146021.8382	95.47	<.0001
pp*bico	2	1745.7434	872.8717	0.57	0.5680
pp*type	3	4912.5133	1637.5044	1.07	0.3679
bico*type	1	3459.1438	3459.1438	2.26	0.1375

Source	DF	Type III SS	Mean Square	F Value	Pr > F
--------	----	-------------	-------------	---------	--------

temp	2	708.51743	354.25872	0.23	0.7939
press	2	2481.12434	1240.56217	0.81	0.4489
pp	3	22013.42592	7337.80864	4.80	0.0045
bico	1	12343.42589	12343.42589	8.07	0.0060
type	1	99111.07651	99111.07651	64.80	<.0001
pp*bico	2	2892.02333	1446.01166	0.95	0.3939
pp*type	3	5406.51597	1802.17199	1.18	0.3250
bico*type	1	3459.14382	3459.14382	2.26	0.137

Dependent variable: Air permeability

Source	DF	Type I SS	Mean Square	F Value	Pr > F
temp	2	699.54749	349.77375	1.23	0.2996
press	2	148.07715	74.03857	0.26	0.7721
pp	3	6403.73838	2134.57946	7.49	0.0002
bico	1	14080.72295	14080.72295	49.38	<.0001
type	1	1058.87608	1058.87608	3.71	0.0581
bico*type	1	192.57213	192.57213	0.68	0.4141

Source	DF	Type III SS	Mean Square	F Value	Pr > F
temp	2	657.35392	328.67696	1.15	0.3218
press	2	55.49302	27.74651	0.10	0.9074
pp	3	1754.69574	584.89858	2.05	0.1148
bico	1	14183.33054	14183.33054	49.73	<.0001
type	1	1206.84966	1206.84966	4.23	0.0435
bico*type	1	192.57213	192.57213	0.68	0.414

Dependent variable: tenacity

Source	DF	Type I SS	Mean Square	F Value	Pr > F
Temp	2	499.5578780	249.7789390	4.05	0.021
press	2	18.6094320	9.3047160	0.15	0.8603
pp	3	429.9717462	143.3239154	2.32	0.0826
bico	1	500.1256046	500.1256046	8.11	0.0058
type	1	592.6305167	592.6305167	9.61	0.0028
bico*type	1	8.2973363	8.2973363	0.13	0.7149

Source	DF	Type III SS	Mean Square	F Value	Pr > F
Temp	2	477.7189604	238.8594802	3.87	0.0255
press	2	13.7318457	6.8659228	0.11	0.8948
pp	3	243.6155278	81.2051759	1.32	0.2762
bico	1	443.0260768	443.0260768	7.18	0.0092
type	1	595.4745066	595.4745066	9.65	0.0027
bico*type	1	8.2973363	8.2973363	0.13	0.7149

VITA

Shaker Gaddam was born in Karimnagar, India on August 30th, 1978. He grew up in Andhra Pradesh state and did his schooling in Karimnagar district. He joined Vignana Intermediate College in Guntur district with his major concentration on mathematics, physics and chemistry. He joined the Osmania University for his Bachelor's degree majoring in textile technology in 1995 and obtained his degree in 1999. In spring 2000, he joined the University of Tennessee, Knoxville. He was working as graduate research assistant while he was studying for his Master's degree in Textile Science.