Presented at CORESTA 2003 (ST 28) and TSRC 2003 (Paper24)

SYSTEMATIC STUDIES ON CIGARETTE PAPER. THE INFLUENCE OF FILLER, FIBRE AND NATURAL PERMEABILITY ON MAINSTREAM ISO YIELDS.

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ABSTRACT

The role of cigarette paper in relation to both the technology associated with the physical manufacture of cigarettes and the resulting smoke yields is an area of interest to the tobacco industry.

The objective of this exercise was to undertake a systematic study on cigarette paper. The experimental format was a statistically based central composite design, in which the permeability range was from 15 to 205 Coresta units, the calcium carbonate filler from 2 to 12 grams per square metre and the fibre 17 to 29 grams per square metre. All the samples were burn additive free and the fibre type used being wood.

Machine made cigarettes were produced with these papers utilising a constant cigarette construction in terms of tobacco column density, blend type, and filter format. Smoking was carried out at standard ISO conditions with analysis for NFDPM, nicotine, carbon monoxide, puff number and static burn rate being undertaken.

Data analysis indicates it is possible to predict the paper physical characteristics and mainstream yields from the papers in question anywhere within the experimental space outlined by the defined variables. Utilising a study of this nature also illustrates the extent to which variation in the paper parameters in question can influence the performance of cigarettes.

Comparison of this approach against the more classical manner of basis weight and percentage chalk inclusion, by which cigarette paper is also defined, will be given. Comment will also be made as to the applicability of the approach, the limitations on the design and interpretations will be explained.

INTRODUCTION.

Cigarette paper plays a significant role in, amongst other factors, the manufacture, visual appearance and objective mainstream yield characteristics of cigarettes.

This paper addresses these three topics in the following manner:-

The manufacture of cigarettes: In this instance the paper physical (mechanical) properties of tensile strength and stretch are used as objective indicators to whether or not a paper can be successfully run on a particular type of cigarette making machine.

The appearance of cigarettes: With the requirement for lower mainstream yields from cigarettes, which is often coupled with the increasing excise duties being payable on cigarettes, the consumer is often concerned with the overall appearance of the product that is being consumed. In respect to cigarette paper appearance this can be objectively assessed by the opacity and whiteness.

The mainstream yields from cigarettes: The regulatory environment in which the cigarette industry operates is becoming more challenging. What the effects are of changing cigarette paper variables on mainstream yields is of interest to cigarette designers. In this instance the ISO mainstream yield characteristics considered are NFDPM, nicotine, CO and puff number.

This paper attempts to address these issues by considering cigarette paper as involving three constructional variables. Namely the quantity of fibre (i.e. cellulose and the fibre types used in this study are all of wood origin), and the calcium carbonate filler ($CaCO_3$/chalk) level. Both of these variables are defined in terms of grams per square metre. The third variable is paper permeability as measured in terms of Coresta units.

This paper does not consider the other often used cigarette paper parameter of burn additive (burning chemical) type(s) and level.

EXPERIMENTAL DESIGN

In order to study the variables described above in a systematic manner and with a view to deriving empirical equations, the approach that has been adopted in this paper is a statistically designed experiment based around a specific design called a Central Composite. The advantage to this form of experimental design is that the number of experiments needed to fully characterise the variables in question is reduced to a minimum. The specific filler, fibre and permeability levels used are given in Table 1 and are represented in pictorial form in Figure 1. As can be seen by reference to this table and figure the design is symmetrically based around a cigarette paper of 23 gsm fibre, 7 gsm filler and 110 CU permeability.

Additionally in Table 1, the target levels of filler, fibre and permeability are given along with the actual levels determined in both the BAT laboratory in the UK and the WFT laboratory in Austria.

Table 1 Central Composite experimental design.

Paper Code	$CaCO_3$ level gsm			Fibre level gsm			Permeability CU		
	BAT	WFT	**Target**	BAT	WFT	**Target**	BAT	WFT	**Target**
1	6.9	7.1	**7.0**	23.4	23.0	**23.0**	14	15	**15**
2	5.2	5.3	**5.0**	20.5	19.7	**20.0**	72	69	**70**
3	4.8	4.9	**5.0**	26.0	26.4	**26.0**	74	71	**70**
4	8.7	8.6	**9.0**	27.5	26.5	**26.0**	67	65	**70**
5	8.5	8.9	**9.0**	20.7	20.2	**20.0**	72	68	**70**
6	7.3	7.2	**7.0**	23.0	22.8	**23.0**	114	106	**110**
7	12.4	12.0	**12.0**	23.1	22.5	**23.0**	113	107	**110**
8	6.7	6.8	**7.0**	29.6	28.4	**29.0**	110	108	**110**
9	7.2	7.1	**7.0**	17.1	17.1	**17.0**	123	113	**110**
10	2.1	2.3	**2.0**	24.1	23.1	**23.0**	118	115	**110**
11	5.1	5.4	**5.0**	20.3	19.7	**20.0**	163	151	**150**
12	8.4	8.9	**9.0**	26.7	26.1	**26.0**	172	160	**150**
13	8.8	9.2	**9.0**	20.1	20.5	**20.0**	184	148	**150**
14	5.2	5.0	**5.0**	26.7	26.3	**26.0**	161	148	**150**
15	7.3	7.0	**7.0**	24.5	23.5	**23.0**	197	218	**205**

CIGARETTES

The paper samples described above were manufactured into cigarettes on a Molins Mark 8 cigarette maker running at 750 cigarettes per minute, and without filter ventilation. The broad cigarette specification is given in Table 2 below.

Table 2 Cigarette construction: - physical and blend details

Cigarette Parameter	**Values and Units**
Physical Measurements	
Cigarette length	84 mm
Tobacco column length	64 mm
Circumference	24.75 mm
Filter Measurements	
Filter length	20 mm
Filter Pressure Drop	70 mm W.G.
Filter material	Cellulose Acetate
Tobacco Rod Weight	690 mg
Blend Chemistry	
Nicotine	2.3 %dwb
Reducing sugars	11.5 %dwb
Total sugars	13.0 %dwb

Note %dwb = % Dry Weight Basis

ANALYSES

The analyses undertaken on the paper samples are as described in various (1) ISO standard methodologies.

All the smoking analyses were carried out on a 20 port Filtrona model 400 linear smoking machine. The analyses undertaken on the products can be looked upon as conventional, i.e. NFDPM, nicotine, CO and puff number, using methodology as for standard machine smoking conditions (2); no minor components were examined. All the products were smoked to the ISO standard butt length of filter + 8mm or overtip + 3mm, whichever is the greater.

RESULTS AND DISCUSSION

With the central composite experimental designs, there are many ways in which the data can be interpreted, a selection of these are given below. Not all the methods are applied to all data sets, but sufficient information is available within the paper such that all the techniques can be applied, to all the data sets, if required.

a) Paper analysis

The data for the paper physical characteristics are recorded in Table 3 below.

Table 3 Paper physical characteristics

Paper Code	**Tensile Strength N/15mm**		**Stretch %**		**Opacity %EU**		**Whiteness %**	
	WFT	BAT	WFT	BAT	WFT	BAT	WFT	BAT
1	14.5	19.5	1.9	2.1	73.1	76.6	90.2	91.3
2	20	18.1	1.3	1.2	67.0	69.1	90.3	91
3	27.5	25.9	1.5	1.6	68.1	70.8	89.8	90.3
4	22.1	20.4	1.4	1.6	72.7	77.9	88.6	89.2
5	16.6	16.2	1.2	1.3	72.8	76.6	92.3	92.3
6	17.7	17.2	1.3	1.2	70.9	74.4	90.2	91.7
7	12.2	12.2	1.0	0.9	76.9	74.8	91.8	90.7
8	23.2	22.5	1.3	1.3	71.1	76.5	87.6	88.2
9	13.9	12.9	1.2	1.3	70.5	71.7	91.3	92.1
10	22.2	21.1	1.5	1.6	61.1	62.1	88.7	89.5
11	17.3	15.6	1.3	1.1	36.9	67.6	87.3	89
12	20.1	18.3	1.2	1.2	72.4	78.0	88.3	89.9
13	13.1	11.5	1.1	1.0	73.0	74.8	89.6	90.7
14	14.8	20.8	1.2	1.4	67.6	72.7	87.4	88
15	15.8	15.4	1.2	1.3	69.1	73.8	86.5	87.2

Note EU = Elrepho Units

One outcome of a study of this nature is that it is possible to produce inter laboratory comparisons (cross checks) for particular parameters, but covering a wide range of cigarette paper variables. This is illustrated for the gsm of filler (chalk/$CaCO_3$) in Figure 2 and good agreement can be seen between the laboratories in question.

By use of a suitable statistical analysis package (such as Minitab or Design Expert) multiple regression analysis can be undertaken on each of the physical characteristic results for the individual papers detailed in Table 3, whilst using the input variables as described for the papers in Table 1. The resulting output is given in Table 4 and in this instance the data used to generate the multiple regression output is that originating from BAT measurements.

Table 4 Multiple regression output for paper physical characteristics relative to the input variables of filler, fibre and permeability.

Multiple Regression Variables	Multiple Regression Coefficients			
	Tensile Strength	Stretch	Opacity	Whiteness
Constant	+ 8.86	+ 20.2	+ 43.85	+ 82.56
Filler	- 0.9	- 0.053	+ 3.71	- 0.38
Fibre	+ 0.89	+ 0.025	+ 0.46	+ 1.16
Permeability	- 0.029	- 0.014	0	$- 3.683\ E^{-3}$
Filler ^{2}	0	0	- 0.13	0
Fibre ^{2}	0	0	0	- 0.031
Permeability ^{2}	0	$+ 4.297\ E^{-5}$	0	$- 2.381\ E^{-4}$
Filler x Fibre		0	0	0
Filler x Permeability	0	0		$+ 5.739\ E^{-3}$
% Fit	91	79	92	90

In Table 4 certain variable, interaction and squared terms are significant for the various paper variables under investigation, thus giving rise to a quadratic equation. These mathematical coefficients for each of the paper physical characteristics in question do not necessarily represent or explain a physical phenomenon for the design variable in question. These equations simply represent a mathematical expression which best fits the results obtained in relation to the variables, of filler, fibre and permeability.

It can be seen that the equations have degrees of fit, with one exception, of 90% plus. Another way of considering the degree of fit is how well the equations generated explain the observed results.

Returning to the original questions posed at the beginning of this paper and considering the 3-dimensional wire frame plots in Figures 3 and 4 it can seen that the tensile strength of the paper is increased with increasing fibre level and decreasing filler levels and permeability. In terms of the physical mechanisms involved, increasing the quantity of fibre increases the level of hydrogen bonding available in the overall paper structure and between individual cellulose chains and hence the tensile strength rises. Conversely increasing the quantity of filler reduces/interferes with the hydrogen bonding and consequently the tensile strength falls. Increasing permeability effectively means opening the fibre structure such that more dilution and diffusion can occur and this in turn also reduces the hydrogen bonding and the tensile strength again falls.

With stretch the results are less clear cut and also a poorer %fit is recorded in the multiple regression output. In part this maybe due to the fact that the range of % stretch results obtained, is numerically low, with this particular set of samples and this makes data interpretation difficult. However the trends recorded and reasoning that can be applied are similar to those for tensile strength.

Figure 5 clearly shows that increasing the quantity of both filler and fibre cause an increase in the opacity of the paper. However there is evidence to suggest in terms of increasing chalk levels that a law of diminishing reward for effort occurs in that the opacity does not increase in a linear manner, i.e. curvature is present in the response surface. Permeability has no effect on opacity.

With respect to whiteness, data interpretation is more difficult, again because the range of results is numerically low, and curvature is present within the response surface.

b) Mainstream yields

The data for the mainstream yields as found in the BAT laboratory are recorded in Table 5 below.

Table 5 Mainstream yield data

Paper Code	Mainstream yields mg/cig			Puff Number	Static Burn Rate mm/min
	NFDPM	Nicotine	CO		
1	18.4	1.49	18.3	8.8	4.03
2	15.7	1.38	13.8	9.2	4.33
3	16.7	1.48	15.8	9.4	4.02
4	15.6	1.33	15.5	8.4	4.91
5	14.6	1.25	13.5	8.2	5.03
6	12.8	1.23	11.6	8.3	4.82
7	13.0	1.21	11.7	8.0	5.09
8	14.5	1.28	14.0	8.7	4.69
9	13.7	1.26	12.0	8.9	4.95
10	15.4	1.43	14.0	9.6	3.99
11	14.3	1.32	12.2	9.0	4.57
12	12.7	1.14	12.0	8.6	5.19
13	11.4	1.11	10.1	8.2	5.16
14	14.8	1.36	13.1	8.9	4.60
15	13.9	1.31	12.4	9.0	5.03

One interpretation of the above data can be undertaken by plotting the individual yield results onto central composite plots such as used to describe the experimental design in Figure 1.

Thus in Figure 6 the NFDPM yields are plotted as a function of the paper parameters in question. When examining data in this format and considering paper permeability as the variable in question there are effectively four pairs of data in which the paper permeability is the only variable that has changed (the filler and fibre levels remaining the same in each of the pairs of results, but different between the pairs of results). Additionally there is one data set of 3 points in which the permeability has changed but with the other paper parameters again remaining constant. The permeability in Figure 6 effectively being the x-axis, examining the data in this manner illustrates that the as the paper permeability increases the NFDPM yield falls and this observation has been reported and explained by others in the past (3-6).

Alternatively rather than considering the results in effectively 3 dimensional space the 4 pairs of data and the single 3 data set can be plotted on a conventional 2 dimensional figure as illustrated in Figure 7.

Finally if required multiple regression analysis can be undertaken and an output of the form given in Table 4 can be obtained if desired.

Considering increasing the filler level on NFDPM yields is also illustrated in Figure 6, using the appropriate pairs etc of data and with the filler level being the z-axis. In this instance increasing the filler level causes a decrease in the NFDPM yield, this can be explained by reference to the static burn rate plot in Figure 8, in which the static burn rate is plotted as a function of the paper parameters. Increasing the level of filler causes an increase in the static burn rate and a corresponding decrease in puff number as illustrated in Figure 9, hence more of the tobacco column is consumed in the inter puff period and a decrease in mainstream NFDPM yield is seen (7).

The converse of this is found when Figure 6 is examined for increasing the level of fibre present in the paper and the mainstream yields increase. Here by reference to Figures 8 and 9 the static burn rate and puff number plots do not give as clear an understanding as is illustrated for the effects of filler. Thus the reasons for this effect are not as clearly understood at present.

Similar comments can be made in relation to examining the nicotine and carbon monoxide yield data.

c) Summarising mainstream yield data and paper physical properties

The results discussed above are summarised in Table 6, where the effects are recorded in a simplified manner.

Thus for example increasing paper permeability reduces the NFDPM yield (↓).
The ↔ sign indicates no clear-cut effect over the range of the variable examined.

The results for % stretch and whiteness are not included for the reasons given earlier.

Table 6 The effect of increasing paper design variables on mainstream yields and paper physical properties

Measured response	**Effect of increasing the paper design variable**		
	Permeability	**Filler level**	**Fibre level**
NFDPM	↓	↓	↑
Nicotine	↓	↓	↑
CO	↓	↓	↑
Puff number	↑	↓	↑
Static Burn Rate	↑	↑	↓
Tensile strength	↓	↓	↑
Opacity	↔	↑	↑

The results illustrate some of the conundrums faced by cigarette and/or paper designers. For example increasing the fibre level, causes an increase in tensile strength characteristics of the paper, which is of interest in relation to the how successfully a paper runs on a cigarette making machine, however this also brings about an increase in mainstream yields. The converse is then true for filler inclusion when increasing the filler level reduces mainstream yields and reduces the tensile strength of the paper.

d) Comparison to defining cigarette paper by, basis weight and % chalk inclusion

The approach described in this paper is to define/characterise cigarette paper in terms of gram per square metre of filler and fibre. A more classical approach is to define cigarette paper in terms of the basis weight (on grams per square metre basis) and % filler inclusion. The use of paper permeability is common to both approaches.

By appropriate transformation of the multiple regression algorithms described in Table 4, it is possible to produce information on a basis weight and % chalk inclusion format. In Figure 10, tensile strength is plotted as a function of % chalk inclusion and it can be seen that as the % chalk inclusion is increased the tensile strength falls. Additionally increasing basis weight generally increases the tensile strength at a given % level of chalk present in the paper.

Thus both approaches give rise to useful information, although however it is should be recognised that in using the more classical approach and stating that a change in basis weight of a cigarette paper is required (for whatever reason) this actually results in changing two variables, the level of filler and the level of fibre. Additionally changing the % filler inclusion in the paper whilst maintaining basis weight, also brings about changes in, the level of filler and the level of fibre. Both of these have been shown in this paper to have effects on the resulting paper physical characteristics and mainstream yields when the papers are manufactured into cigarettes. The use of basis weight becomes further complicated when burn additive is included in the paper, in this situation a change in basis weight results in a change to three variables, the gsm of filler, fibre and burn additive.

The two approaches, described above, for examining/characterising cigarette paper can also be applied to the resultant mainstream yields if required.

CONCLUSIONS

Statistically based experimental designs can be applied to cigarette paper and in this instance the variables used have been paper permeability plus filler and fibre expressed on a gram per square metre basis. It should be noted that this represents a significant quantity of work, both from the paper and cigarette manufacturer's standpoints.

It is then possible to explain and predict the resultant paper physical properties in relation to these variables. Namely that increasing the quantity of fibre increases the mechanical properties of the paper and the opacity. Increasing the level of chalk decreases the mechanical properties of the paper and increases the opacity. These mechanical effects can be rationalised by considering hydrogen bonding that occurs between cellulose molecules in the fibres. Increasing permeability decreases the mechanical properties of the paper and has no effect on the opacity.

When cigarettes are manufactured from these papers and analysed for mainstream smoke yields the results indicate that increasing the quantity of fibre causes an increase in all mainstream yields, whereas increasing the quantity of chalk reduces all the mainstream yields. In relation to the effect of filler these observations can be explained in part by examining the static burn rate of the products.

The approach described in this paper in terms of describing the cigarette paper in terms of the variables of filler and fibre on a gram per square metre basis can be looked as being a complimentary approach to the more classical approach of defining a paper in terms of basis weight and % filler inclusion. The advantage to the approach described in this paper is that the variables are looked on as being independent, whereas in the classical approach they are inter related or confounded.

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Figure 1. Schematic of Central Composite Experimental Design

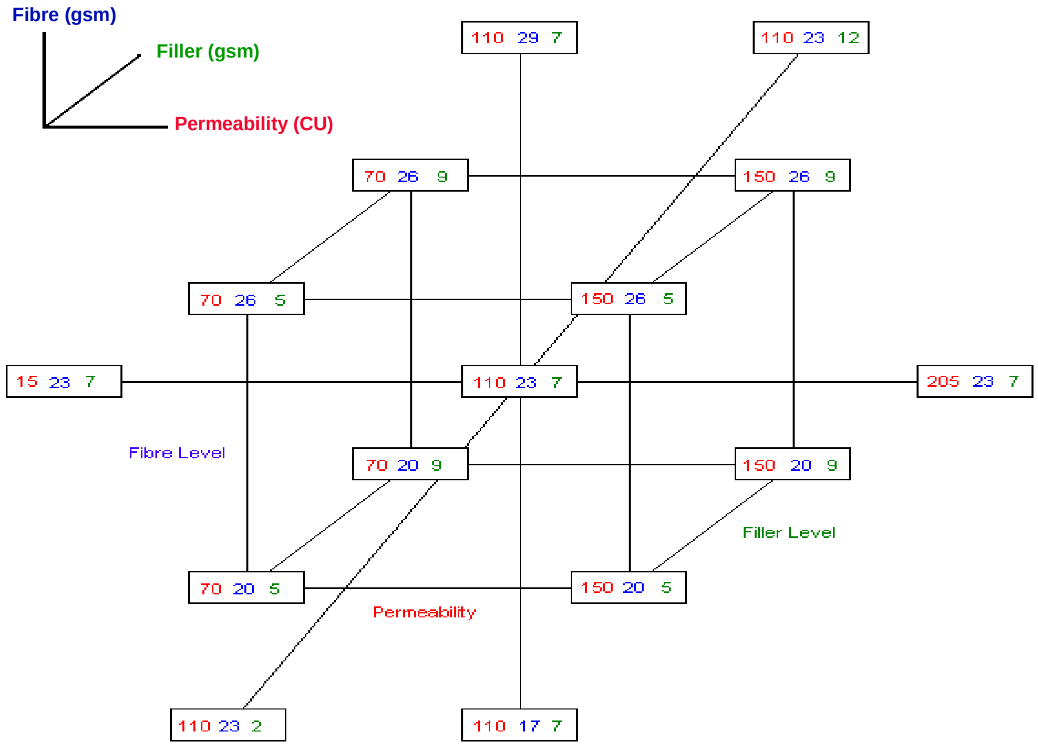

Figure 2. Comparison of $CaCO_3$ level in Paper between BAT and Wattens Laboratories

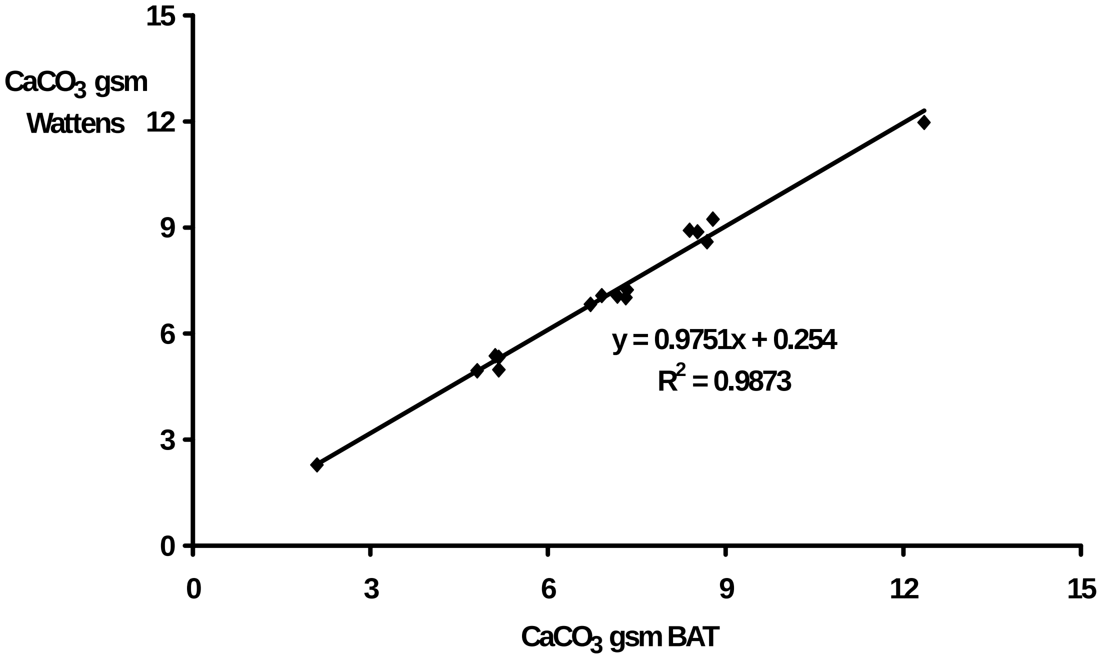

Figure 3. Tensile Strength as a function of Filler and Fibre

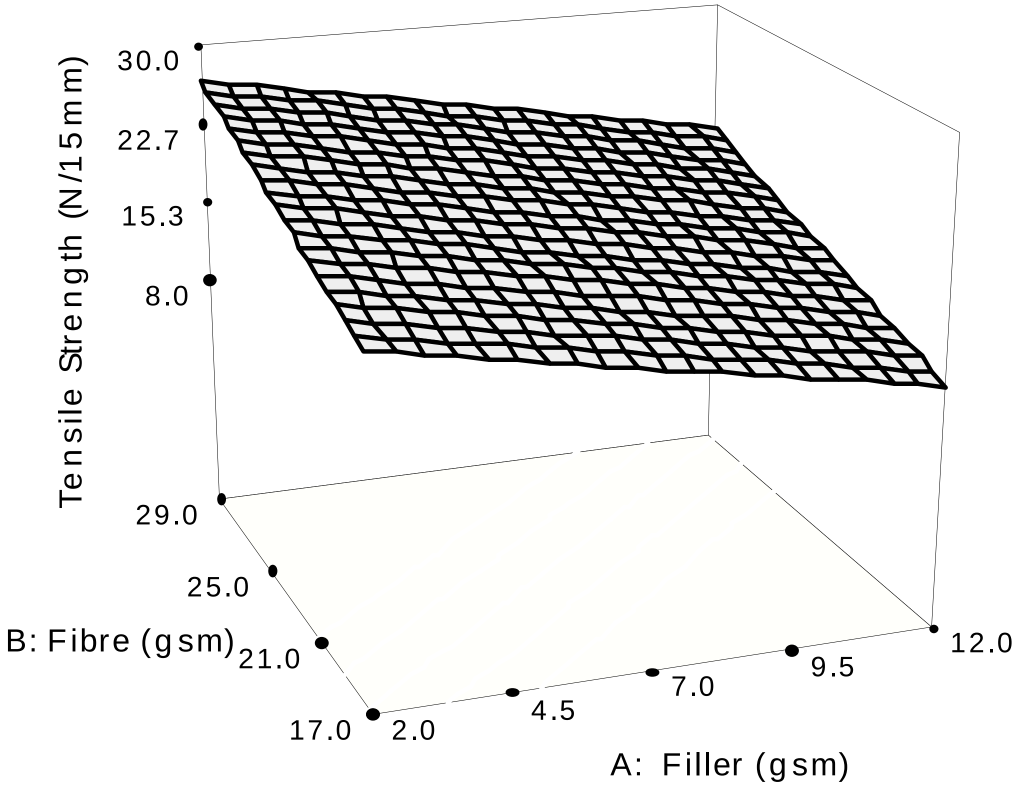

Figure 4. Tensile Strength as a function of Permeability and Fibre

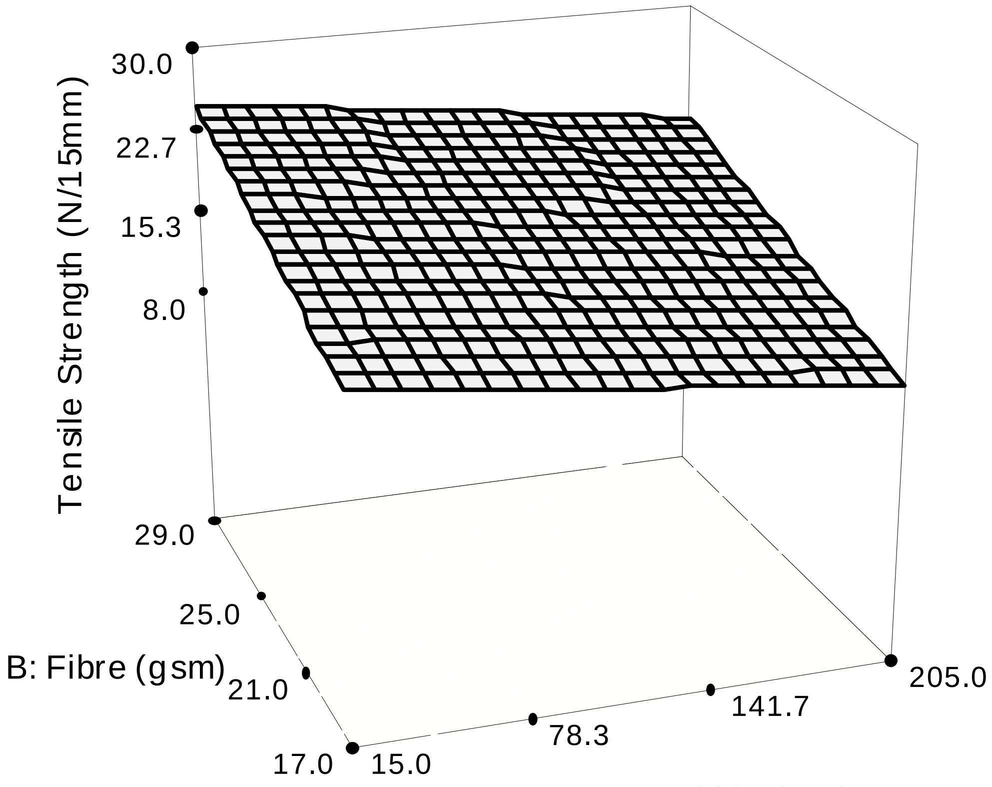

Figure 5. Opacity as a function of Filler and Fibre

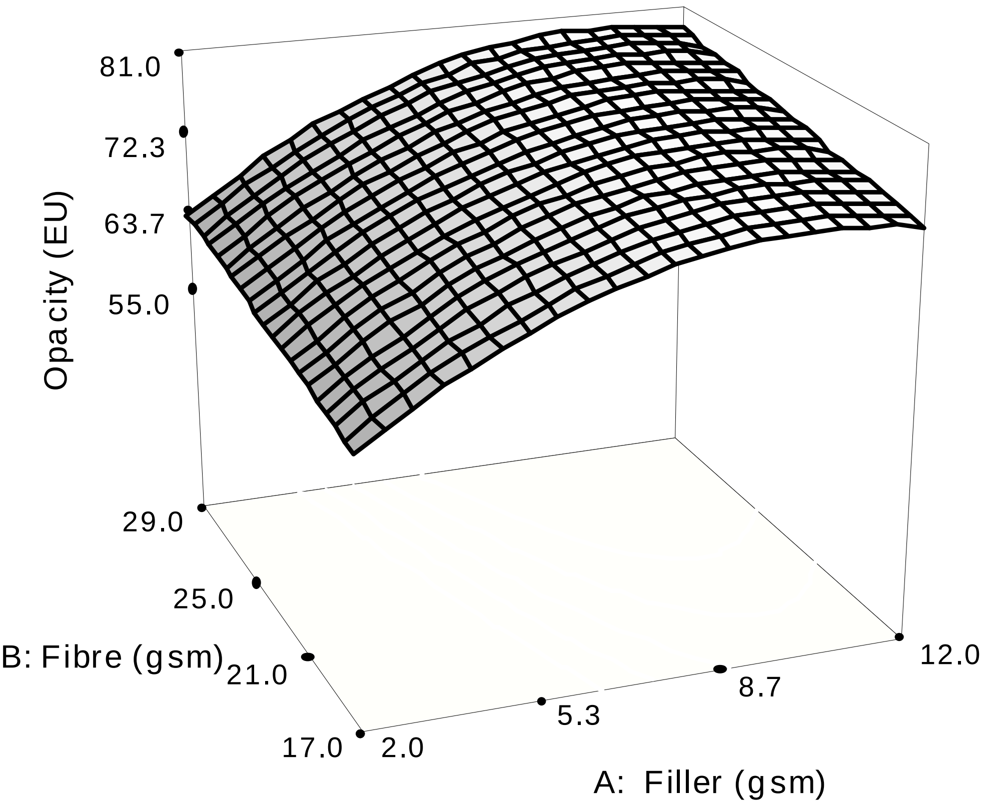

Figure 6. NFDPM yields as a function of Filler, Fibre and Permeability

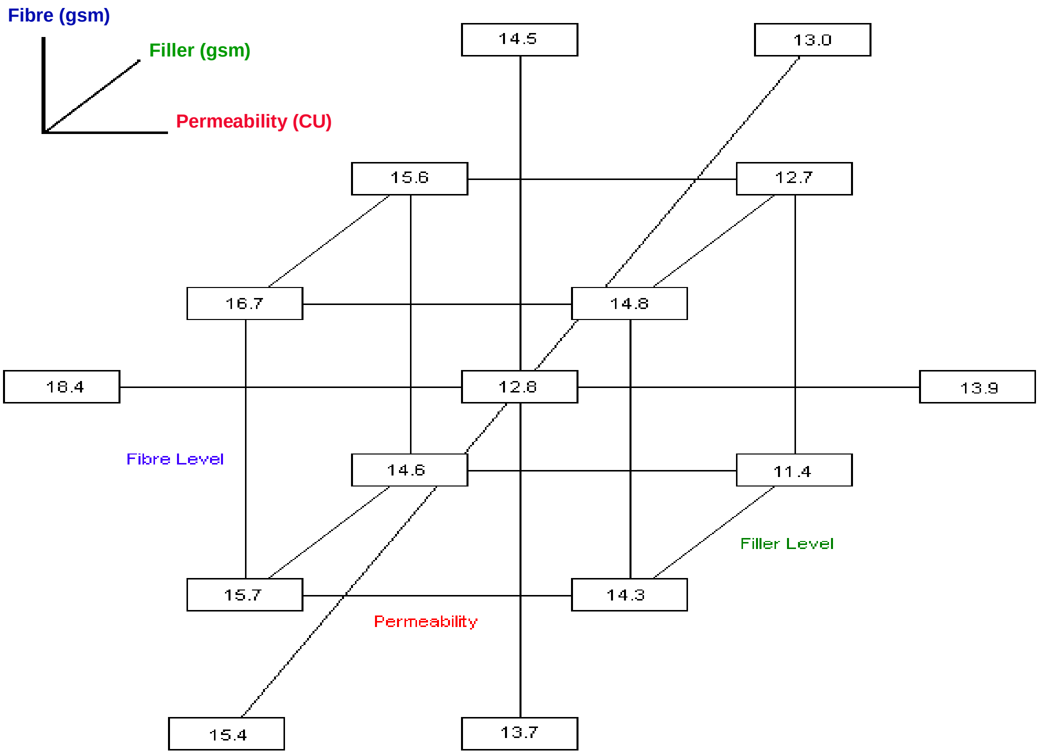

Figure 7. Mainstream NFDPM Yield as a function of Permeability, Filler and Fibre

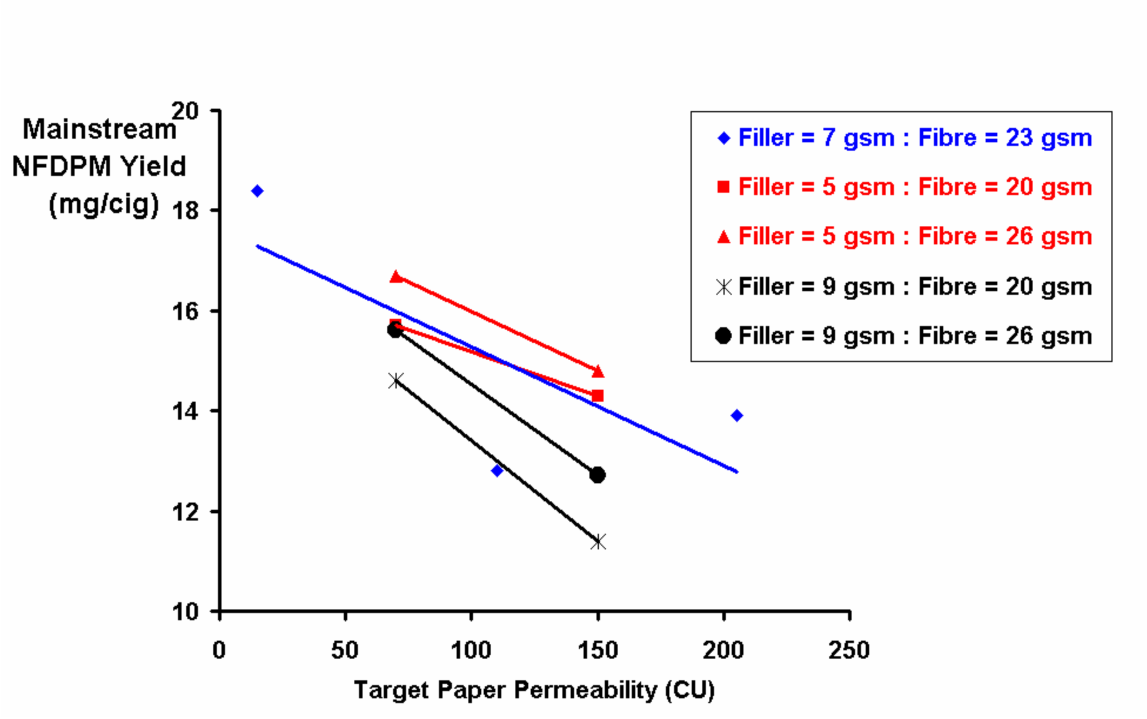

Figure 8. Static Burn Rate as a function of Filler, Fibre and Permeability

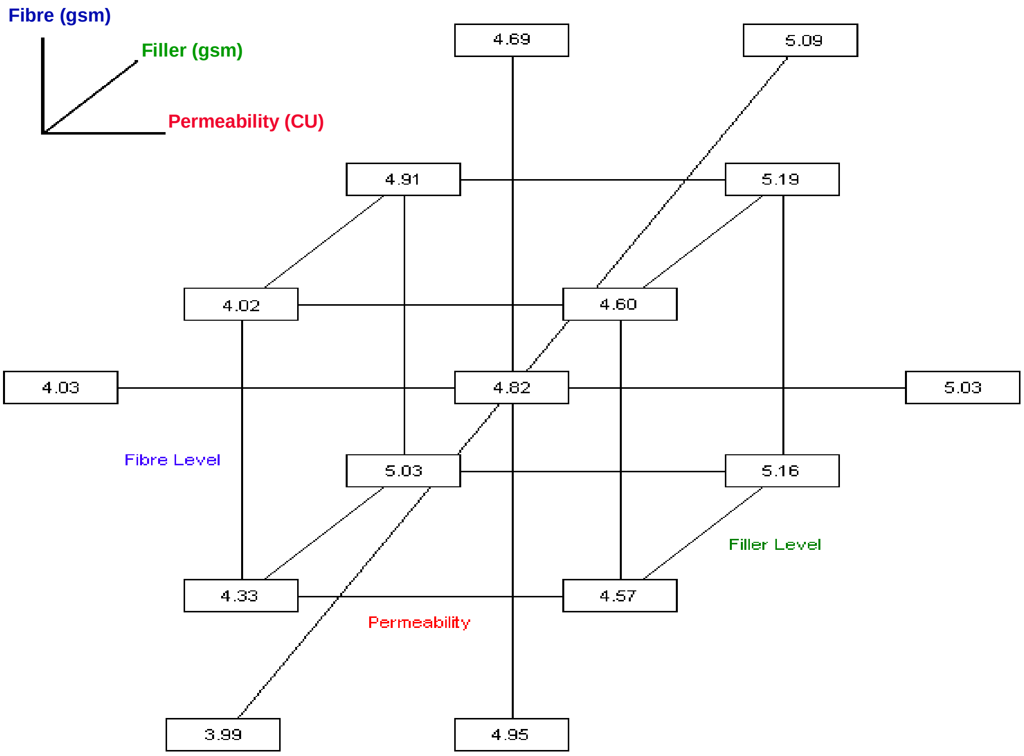

Figure 9. Puff Number as a function of Filler, Fibre and Permeability

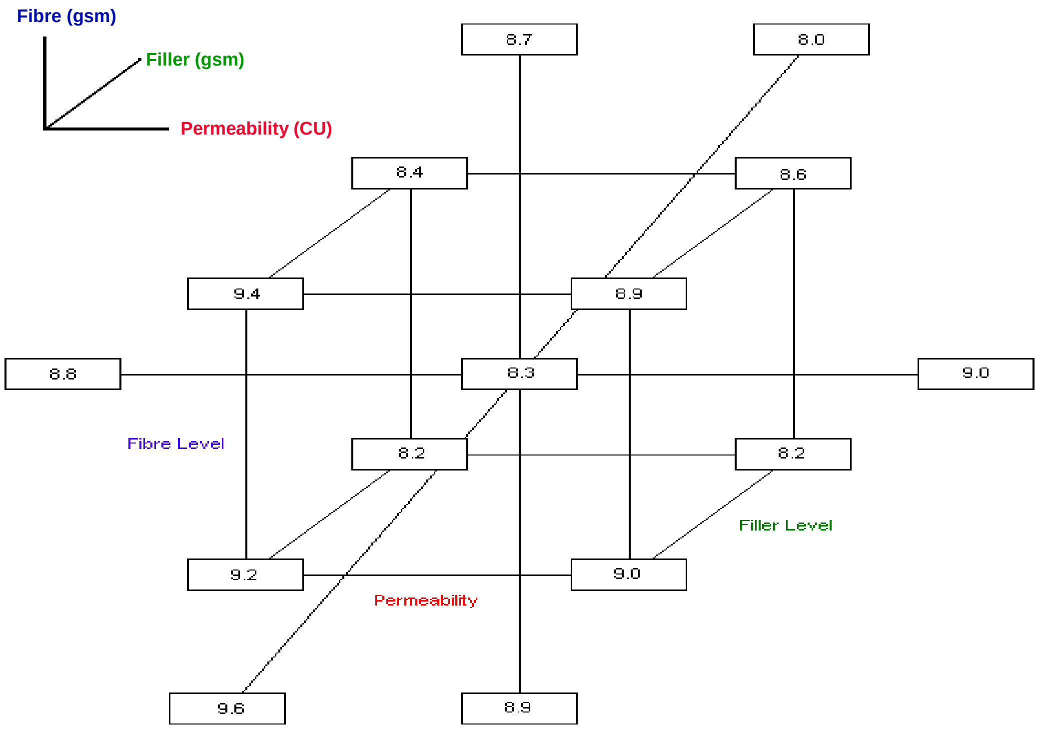

Figure 10. Paper Tensile Strength as a function of % $CaCO_3$ and Basis Weight