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PRE-HYDROLYSIS PULPING PROCESS OPTIMIZATION WITH MULTIPLE RESPONSE SURFACE MODELLING

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ABSTRACT

Three properties of dissolving pulp namely lignin, viscosity and the α-cellulose were investigated. A laboratory experiment for the dissolving wood pulping process was conducted on nine *Eucalyptus* genotypes: *Edunnii, Esmithii, Egrandis, Macarthurii, Emearnsii, Enitens, GCG438, GUA380* and *GUW962*. Repeated measurements were taken at each of the six processing stages for the changes in lignin, viscosity and the α-cellulose. A response surface approach was used to select the best genotype for each property and further application of desirability analysis to identify the genotype that simultaneously gives the best results for the three properties. The predictive models and associated statistical tests proved that all the nine genotypes were capable of producing the optimal results (>95.55% α-cellulose) although a few were at the thresholds of the feasible region. The optimisation process also revealed that the genotype Emearnsii possesses the most desirable properties for the α-96 cellulose product output and Enitens consistently produces results within the desired range. The use of simultaneous desirability functions indicated that the overall product quality characteristics for lignin, viscosity and the α-cellulose can be improved by steadily excluding the most resistant genotypes to lignin reduction, especially *Edunnii* and *Esmithii*.

KEYWORDS: Pulp, *Eucalyptus* genotypes, variable transformation, desirability and ridge analysis

INTRODUCTION

The three main desirable chemical properties in the wood dissolving pulp are α-cellulose, viscosity and lignin (Tappi 1994, 1999, 2000, 2002). Their optimisation in the product mix improves the quality of the production output. The α-cellulose indicates undegraded and a higher molecular weight in pulp. The lignin removal provides information about the performance of the process since it determines the hardness, bleachability and other pulp properties. Measuring viscosity gives an average degree of cellulose polymerisation.

The timber supplied to the mills exists in different species that are likely to behave differently at the processing stage. The differences could be related to their genotype (Melesse and Zewotir 2013, 2015); or the rate of change of the chemical properties across the processing stages and genotype (Bodhlyera, et al. 2014, 2015). Their studies, however, did not take into account the product quality optimisation. In order to improve the cellulose product quality, Kristina (2005) also considered a multivariate characterisation and analysis of the reactivity and spectroscopic properties in dissolving pulp revealing the short cellulose chains and low molecular weight in high reactivity pulp. Nevertheless, the focus was on the viscosity alone of which it is not the sole chemical property for determining the α-cellulose product. In all these studies, the optimal effect of each genotype to the product quality was not factored in despite their different genetic make ups. Reluctantly, the same production line and common chemical treatment is used across the board for all the genotypes, yet some of the genotypes require less attention than the others. The key question to this study was which genotypes need more attention and are the best for each of α-cellulose, viscosity and lignin or their combinations.

This study endeavoured to stretch the horizons of the current knowledge on dissolving wood pulp process by refining the use of or raw materials (genotype ingredient mix) enhancing the economic utilisation of other resources such as chemical requirements, time and equipment. Being fully equipped with the knowledge of α-cellulose, viscosity and lignin optimisation in relation to the Eucalpytus genotypes genetic makeup allows proper production, planning and scheduling as specific types of timber can be transported and stored accordingly, in preparation for processing depending on customer specifications and production targets. Appropriate statistical techniques have been developed such as response surface modelling in the optimisation procedure to become the lens through which the chemical properties' behavioural patterns can be viewed. This further call for the application of the desirability analysis which is capable of answering more investigative questions of interest for the dissolving wood pulp process improvement, oiling the wheels of production in the forestry and related industries.

Response surface methodology (RSM)

For a 3D visualisation of the data, the use of a response surface approach gives a better insight to determine the optimum region of the factor space in which the operating conditions are met (Montgomery 1991). Determining the factor settings for the response is useful depending on the product or process requirements (Relia Soft Corporation 2015). Liu (2012) indicated that the response variable must be a quantitative continuous variable to model the curvature effects in the scientific areas using the response surface methodology; hence both predictor variables stage and genotype were coded accordingly. The nine genotypes were coded based on their average resistance to lignin reduction, with the most resistant as 1 and the least resistant as 9: 1-*Edunnii*, 2-*Esmithii*, 3-*GCG438*, 4-*Egrandis*, 5-*GUA380*, 6-*GUW962*, 7-*Macarthurii*, 8-*Emearnsii* and 9-*Enitens*. The processing stages were coded according to their sequence on the production line, that is, 1 to 6 for the six sages. The response surface with polynomials of higher degree is given by:

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i}\sum_{\substack{j \\ i<j}} \beta_{ij} x_i x_j + \varepsilon \qquad (1)$$

Where the regression coefficients are obtained by the method of least squares and x_i, $i = 1...k$ are factors.

The response variable may be transformed to improve model accuracy using the relationship where y is the original variable and Y the transformed (Box and Draper 1987). The canonical analysis procedure is used to investigate the shape of the predicted response surface (SAS Visual Analytics 7.1, 2014). In matrix notation (Eq. 1) can be represented as $\hat{\mathbf{y}} = \hat{\beta}_0 + \mathbf{x}'\mathbf{b} + \mathbf{x}'\mathbf{B}\mathbf{x}$ where the vector **b** represents the first-order regression coefficients and **B** is a symmetric matrix. Concentric circles of radius r_i in the ridge analysis search are related to the predictor variables as $\sum_{i=1}^{k} x_i^2 = r_i^2$ which are equivalent to $\mathbf{x}'\mathbf{x} - r^2 = 0$. The set of equations satisfies:

$$\begin{array}{ll} \textit{Maximise(or minimize)} & \hat{y} = \hat{\beta}_0 + \mathbf{x}'\mathbf{b} + \mathbf{x}'\mathbf{B}\mathbf{x} \\ \textit{Subject to} & \mathbf{x}'\mathbf{x} - r_i^2 = 0 \;\;, \textit{for } i^{th} \textit{ circle} \end{array} \quad (2)$$

Desirability analysis

The desirability analysis simultaneously optimizes the multiple response surface equations by employing the overlaid contours and the desirability functions. The feasible region is the area that is satisfied by all the responses. A desirability function transforms the response variable to d_i where $1 \leq i \leq R$ with $0 \leq d_i \leq 1$ and R being the number of response surface equations to be simultaneously optimized. Irrespective of the shape, the value $d_i = 0$ represents a completely undesirable response and $d_i = 1$ being the most desirable one. The functions differ depending on the response objective, that is, maximisation goal reached in the direction of the upper limit (U), minimisation towards the lowest value (L) or on target (T) to be achieved. Derringer and Suich (1984) supposed that if the R response surface equations are denoted by $\hat{y}_i$, three forms of the desirability functions corresponding to the type of the optimization goal are given as follows:

$$d_i^{Max} = \begin{cases} 0 & \hat{y}_i < L \\ \left(\frac{\hat{y}_i - L}{U - L}\right)^s & L < \hat{y}_i < U \\ 1 & \hat{y}_i > U \end{cases} \quad d_i^{Min} = \begin{cases} 1 & \hat{y}_i < L \\ \left(\frac{\hat{y}_i - U}{L - U}\right)^s & L < \hat{y}_i < U \\ 0 & \hat{y}_i > U \end{cases} \quad d_i^{Target} = \begin{cases} 0 & \hat{y}_i < L \\ \left(\frac{\hat{y}_i - L}{T - L}\right)^{s_1} & L < \hat{y}_i < T \\ \left(\frac{\hat{y}_i - U}{T - U}\right)^{s_2} & T < \hat{y}_i < U \\ 0 & \hat{y}_i > U \end{cases} \quad 3)$$

To set up a desirability criterion that is either easier or more difficult to satisfy, it depends on the chosen values of s, s_1 and s_2. The Eq. 3 give the individual desirabilities for each response variable. The overall desirability function for the R equations is a geometric mean of the individual desirability functions given by $D = \left(\prod_{i=1}^{R} d_i\right)^{1/R}$. In addition to the d_i property, D = 0 if any one $d_i = 0$ and consequently, the overall desirability is also unacceptable.

MATERIALS AND METHODS

Laboratory procedure

The process can be classified as delignification, bleaching and finishing. Delignification is acid bisulphite pulping and takes place in a digester whereby wood chips are circulated in bubbling SO_2MgO slurry to produce cooking liquor. Bleaching and finishing entail the bleaching of the O_2 delignified pulp samples to a target of 96α grade in the following sequence: (1)-Raw pulp, (2)-Delignification, (3)-Bleaching, gamma cellulose and lignin removal with CIO_2 treatment, (4)-Bleaching, gamma cellulose and lignin removal with NaOH treatment, (5)-Bleaching, gamma cellulose and lignin removal with CIO_2 treatment and (6)-Peroxide stage (Bodhlyera, et al. 2014, Tappi 1994, 1999, 2000, 2002).

The data

The experimental results involved nine genotypes *Edunnii*, *Esmithii*, *Egrandis, Macarthurii, Emearnsii, Enitens, GCG438, GUA380* and *GUW962* from which the three chemical properties (viscosity, lignin and α-cellulose) of interest were recorded as the response variables. A randomisation process was employed that used 16 different trees from the nine genotypes and these were sampled from eight different climatic conditions (site qualities) ranging from warm to cold. The different samples (258 observations) were also grouped into three categories which were basically the different bleaching conditions (Tab. 1). *Esmithii* had two of its cold weather samples allocated to bleaching condition C and similarly, two of the *GUW962's* site quality 1 samples randomly assigned to the bleaching condition B. Hence, each sample was characterised by its genotype, site quality, tree and the bleaching condition. Repeated measurements of the chemical properties were then taken from each sample at every stage (six times) to come up with the data set of 258 observations.

Tab. 1: The randomisation process for the sample observations.

		Bleaching Condition			
Genotype	Site Quality (SQ)	A	B	C	Total
Edunnii	SQ2	6	6		**12**
	SQ3	6	6	6	**18**
	SQ4 Warm weather	6	6	6	**18**
	SQ5 Cold weather	6	6	6	**18**
	SQ5 Warm weather	6	6	6	**18**
EGrandis	SQ2			6	**06**
	SQ3	6	6	6	**18**
	SQ4 Warm weather			6	**06**
Emearnsii	SQ5	6	6	6	**18**
Enitens	SQ3	6	6	6	**18**
ESmithii	SQ3	6	6	6	**18**
	SQ3 Cold weather	6		12	**18**
GCG438	SQ2	6	6	6	**18**
GUA380	SQ1	6	6	6	**18**
GUW962	SQ1		12	6	**18**
Macarthurii	SQ3	6	6	6	**18**
	Total	**78**	**84**	**96**	**258**

RESULTS AND DISCUSSION

Data analysis software

The data exploration and analysis were conducted in Statistical Analysis System (SAS 9.3); John's Macintosh Program (JMP 12); Design Expert 9; SPSS 23 and Ms Excel. Each response variable was regressed by the two predictors (stage and genotype). The models were then used to plot the response surfaces and contours; explore the experimental region using ridge analysis and formulating the desirability function to search for the optimal solution.

Results of data exploration

The exploratory results of each response against the genotype and processing stage show that *Edunmii*, *Emearnsii*, *Esmithii* and *Macarthurii* genotypes have low *α*-96 cellulose levels in Stage 6. At the beginning of the process, *Emearnsii* seems to have started with a very high *α*-96 Cellulose level but eventually had the least output. On the other hand, *Edunnii*, *Emearnsii* and *GUW962* show that they cannot be mixed with the other genotypes since they have much lower levels of viscosity at the finishing stage. *Enitens* is also the first to reach very low lignin levels followed by *GUW962* which also started low. *Edunmii* and GCG438 are the most resistant to lignin reduction as they trailed off to reach the lowest levels in the final stage of the dissolving pulp process.

Results of the response surface models

α-96 cellulose

The Box Cox transformation suggested that Y_c , the transformed value of *α*-96 cellulose be given by $Y_c = y_c^{-3}$ to improve the prediction power as shown in Fig.1.

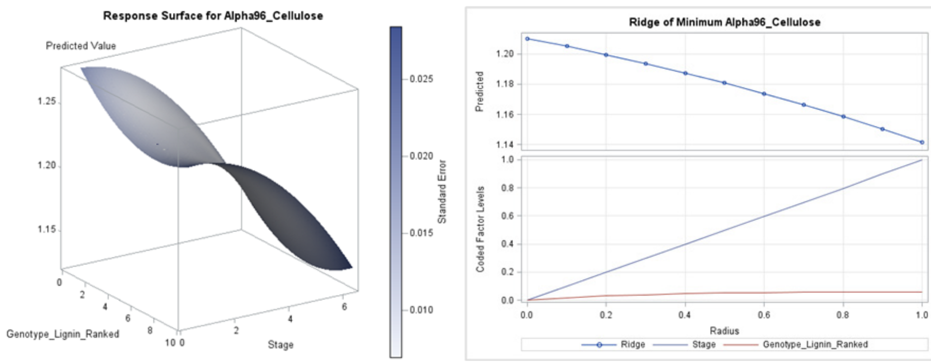


Fig. 1: Response surface and ridge analysis of the transformed α-96 cellulose.

The transformation improved the variation in the response explained by the predictors from 0.1877 to 0.2133 and root mean square error (RMSE) of the transformed variable was down to 7.0548 x 10^{-2} from 0.4403. The *α*-96 cellulose was modelled by the linear (p-value < 0.0001) component only of the variable stage. Kutner et al. (2005) explained that for practical purposes, most of the information is captured by the second order model hence higher order models were not considered. The eigen values had different signs, hence the stationary point was a saddle and also outside the given data range. The stage value of 0.6406 was not in the predictor variable range (1 to 6). At this point *Macarthurii* was the best candidate for the maximisation of the *α*-96 cellulose. The maximum absolute eigen value (0.0205) corresponded to the maximum eigenvector (0.9992) for the stage indicating that the surface curved more along stage than it does along the genotype axis. The ridge analysis (based on transformed values) shows that from the centre (Stage = 3.5; Genotype = *GUA380*), the *α*-96 cellulose values decrease as the process approaches the final stage but the genotype remained the same.

Viscosity

The Box Cox transformation produced in JMP used the sum of the squared errors SSE of prediction instead of ln (SSE) which is just a scaled version (Box et al. 1987) and transformed viscosity to $Y_v = y_v^{-0.29}$ giving rise to improved predictions in Fig. 2.

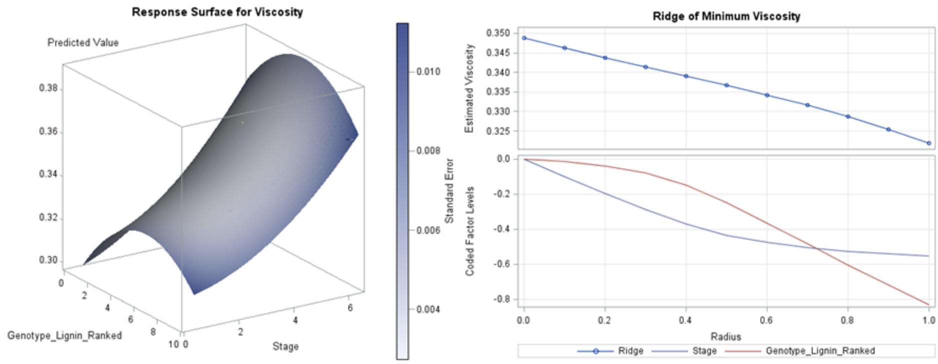


Fig. 2: Response surface and ridge analysis of the transformed viscosity.

This improved the RMSE from 13.0555 down to 0.0278 with a slight positive change on the r^2 = 0.3509 (6% increase). The significant (p-value < 0.0001) fitted model included both linear (p-value < 0.0001) and quadratic (p-value < 0.0012) terms. Stage (p-value < 0.0001) and genotype (p-value < 0.0005) were both significant factors in the viscosity model. Since the coded factors are scaled (+1 for highest levels and -1 for lowest levels), the coded model coefficients can be used for identifying the relative impact of the factors by simply comparing the factor coefficients. The coded parameters indicated that the intercept (0.3489) was the most influential term followed by stage (0.0265) and the genotype quadratic term (-0.0158). The optimal point for viscosity was also a saddle at genotype code 6 (*GUW962*) with the stage number (-1.0644) being outside the experimental region. The transformed minimum viscosity value was 0.3251 (actual = 48.1863). The largest eigenvector (0.9990) corresponded to the maximum (0.0158) absolute value of the eigen values suggesting that the surface curves more along the genotype axis than it does along the stage axis. The genotypes that are more resistant to lignin reduction tend to have lower transformed viscosity (higher for actual) values.

Lignin

The fitted model for lignin is shown in Fig. 3. The lignin max to min ratio (63) was greater than 10 and the Box Cox power transformation suggested that the lignin response values be

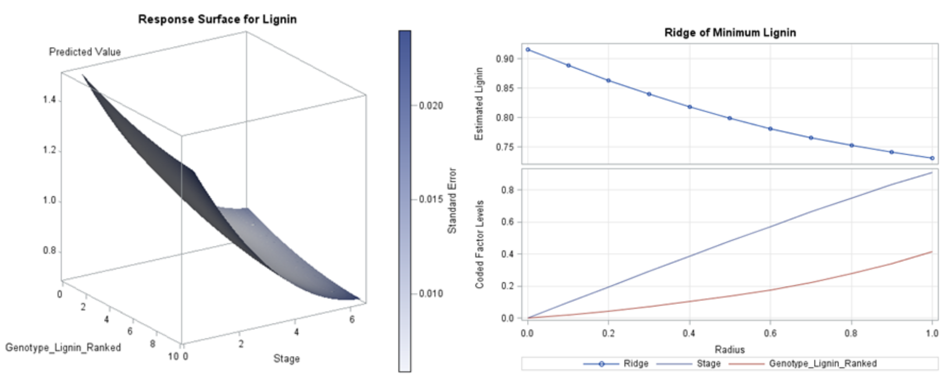


Fig. 3: Response surface and ridge analysis of the transformed lignin.

transformed to $Y_l = y_l^{0.21}$. The lignin model was significant (p-value < 0.0001) with the model fitness indicating 92% of the lignin reduction variation explained by the regressors.

The errors (RMSE = 0.0593) were very small adding more confidence to the credibility of the lignin model adequacy. The significant model for lignin was of the second order, with both the linear (p-value < 0.0001) and quadratic (p-value < 0.0001) components were significant as well. In addition to the intercept, stage, genotype and the quadratic term for stage were also significant with all having p-values < 0.0001. The canonical and ridge analysis had all the eigen values positive suggesting that the stationary point was a minimum. The solution (Stage = 6.6976, Genotype =10.4885 and actual Lignin = 0.18010) shows that both predictor values were outside the data range. The curvature was more defined on the stage factor because of the greater eigen value (0.1068) that corresponded to the larger eigenvector (0.9996) of the regressor stage (Fig. 3.). There is a negative correlation between factor levels and the amount of lignin that remained in the α-96 cellulose output. The highly resistant genotypes have more lignin content and the least resistant genotypes having much lower content.

Parameter estimates of the transformed models

Tab. 2 gives a summary of the transformed parameter estimates for predicting the three responses (lignin, viscosity and the α-cellulose). Great care should be taken when interpreting the transformed variables especially when the power transformation is negative as this reverses the objective function.

Tab. 2: Parameter estimates of the transformed responses.

Response:		*α-96 cellulose*		*Viscosity*		*Lignin*	
Transformed (Y) objective:		*Minimize*		*Minimize*		*Minimize*	
Box Cox transformation:		$Y_c = y_c^{-3}$		$Y_v = y_v^{-0.29}$		$Y_l = y_l^{0.21}$	
Parameter	DF	Estimate	Pr > |t|	Estimate	Pr > |t|	Estimate	Pr > |t|
Intercept	1	1.2790	<.0001*	0.2935	<.0001*	1.6265	<.0001*
Stage	1	0.0023	0.8623	0.0037	0.4774	-0.2337	<.0001*
Genotype	1	-0.0131	0.0903**	0.0114	0.0002*	-0.0279	<.0001*
Stage²	1	-0.0033	0.0627**	0.0011	0.1014	0.0171	<.0001*
Genotype*Stage	1	0.0003	0.7611	-0.0002	0.5748	0.0005	0.5561
Genotype²	1	0.0010	0.2001	-0.0010	0.0010*	0.0012	0.0642*
Raw response (y) objective :		*Maximize*		*Maximize*		*Minimize*	

Sig.: * 0.05; **0.10

With the exception of the intercept, Tab. 2 parameter estimates are herein interpreted according to the objectives of the raw responses. For example, a negative coefficient of a Box Cox transformed variable would actually mean an increase in the raw response and vice versa. The coefficients and objective function for the positive Box Cox power transformations remain unchanged and so does the interpretation too as in the case of lignin. All the models were capable of estimating the responses in the raw pulp (significant intercepts, p < 0.0001) and lignin significantly (p < 0.0001) decrease with each stage of processing. The linear components of the genotypes significantly (at 10% level of significance) contribute to response changes where both viscosity and lignin generally decrease whilst cellulose increases when the least resistant genotypes are added to the model. Similarly, cellulose increases with the quadratic factor of stage whereas the quadratic factor of genotype increases both the viscosity and lignin contents. Lignin also decreases at each processing stage and when the genotypes less resistant to lignin removal are added. The interaction between stage and genotype has been proved to be statistically insignificant on all the responses.

Ridge analysis of the raw responses

For r =1 (Tab. 3), GUA380 has the maximum α-96 cellulose output value of 95.6883%. During the steepest descent, in stages 3 to 6, GUA380 was dominant in giving the best results.

Tab. 3: Estimates of ridge and steepest descent optimal search for the raw responses.

Raw response (y):	*α-96 cellulose*			*Viscosity*			*Lignin*		
Objective :	*Maximize*			*Maximize*			*Minimize*		
Coded Radius (r)	Stage	Genotype	Raw Responses (%)	Stage	Genotype	Raw Responses (*cP*)	Stage	Genotype	Raw Responses (*k-number*)
0.0	3.50	5.00	93.8392	3.50	5.00	37.7610	3.50	5.00	0.6587
0.1	3.75	5.07	93.9714	3.25	4.94	38.7477	3.74	5.09	0.5697
0.2	3.99	5.12	94.1144	3.01	4.84	39.7201	3.99	5.18	0.4961
0.3	4.24	5.16	94.2685	2.78	4.68	40.6819	4.23	5.29	0.4353
0.4	4.49	5.18	94.4341	2.57	4.41	41.6493	4.47	5.41	0.3851
0.5	4.74	5.20	94.6116	2.41	4.01	42.6580	4.70	5.55	0.3437
0.6	4.99	5.22	94.8012	2.31	3.54	43.7579	4.93	5.71	0.3096
0.7	5.24	5.23	95.0033	2.24	3.06	44.9943	5.16	5.90	0.2816
0.8	5.49	5.23	95.2183	2.19	2.59	46.4014	5.38	6.11	0.2587
0.9	5.75	5.24	95.4465	2.15	2.12	48.0086	5.58	6.36	0.2401
1.0	6.00	5.24	95.6883	2.12	1.67	49.8438	5.78	6.66	0.2251

Increasing the radii up to 1.5 (extending the experimental region) from the centre (3.5; 5) improved results to 96.7997% and GUA380 still gave the best α-96 cellulose results. The standard errors of the estimates were very small giving much confidence to the predictions. The max to min ratio for the viscosity original values was 5.11, with the r^2 = 0.33 and RMSE = 13.0555. The maximum viscosity value of 49.8438 was reached in Stage 2 with genotype code 2 (*Esmithii*) being able to achieve such a high value. Increasing one more circle (r = 1.1) would still be in the experimental region and improved the viscosity results to 51.9398cP in Stage 2. The improved results were produced by *Edunnii* (genotype code 1), the most resistant genotype to lignin reduction. The SAS default radius (r = 1) for the search of lignin factor settings shows that the steepest descent provides the minimum lignin content of 0.2251 in the final stage and from genotype code 7 (*Macarthurii*). Likewise, stretching the boundaries of the search region improved the lignin results to 0.2131 and still from *Macarthurii* in the final stage again.

Results of the desirability analysis

Optimal solution

The three response surface models for α-96 cellulose, viscosity and lignin were used to derive the desirability functions using JMP and the plot of contours in Fig. 4. The dotted points along each contour show the side on which the transformed values are going up (down for the actual or raw data except lignin). The feasible region (white) provides the set of constraints (Tab. 4) or the required and ideal response limits to produce the best α-96 cellulose output. To emphasize the importance of the goals for the three responses, α-96 cellulose was assigned to carry more weight (0.7) since it is the final product, followed by lignin (0.2) as the process aims to remove it without damaging the molecular structure (viscosity giving it 0.1 weight).

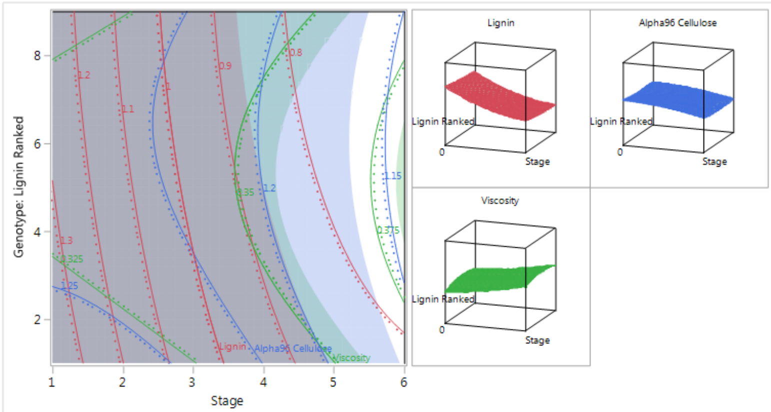


Fig. 4: Overlaid contours and the overall optimum response region.

Tab. 4: Response constraints.

	Lignin		Viscosity		Alpha 96 cellulose	
Weight	0.2		0.1		0.7	
Objective	Min	Min	Min	Max	Min	Max
	Transformed	Actual	Transformed	Actual	Transformed (x10^-6)	Actual
Lower Limit	0	0	0.3805	28.0	1.1664	95
Target	0.7474	0.25	0.3677	31.5	1.1303	96
Upper Limit	0.8645	0.50	0.3566	35.0	1.0625	98

The optimal region based on these constraints shows that all the genotypes are capable of hitting the α-96 cellulose output target of 95.55% within the specified constraints and still show room for improvement. The weakest genotype within the feasible region is Genotype 5 (*GUA380*) which cannot exceed 95.55% cellulose due to viscosity restrictions as it quickly reaches the lower limit (28cP), followed by genotypes 4 (*Egrandis*) and 6 (*GUW962*) which also yield good α-96 cellulose but with the molecular weight very close to the viscosity lower limit. Any further attempt to increase α-96 cellulose percentage will quickly bring down the viscosity level to below 28cP. An increase in α-96 cellulose level will result in the product output with a much lower degree of polarisation. *Edunnii* is much better for high viscosity and Macarthurii good for lower lignin content. The two genotypes proved to have the capacity to exceed 95.55% α-cellulose. Based on the exploration of the optimal region boundaries, the best candidates for individual responses that are also consistent in the optimal region are *Edunnii* and *Macarthurii*.

Results of the refined feasible region

An exploration of the optimum region without the application of sophisticated mathematical formula considered each response's best values along the boundary that is giving the best results of α-96 cellulose (highest weight). The best results for each genotype were recorded per response and later ranked or scored 1 (poor) to 9 (Excellent), the best achievement as the objective requires. Each genotype ended up having three scores from lignin, viscosity and α-96 cellulose output. Summing up of these scores provided the overall score for each genotype, with the highest score representing the best optimal genotype. The scores were also expressed as percentages of the maximum achievable score (27) to provide a better measurement unit of how capable is the genotype in giving the most desirable results. The highest points were scored by *Emearnsii* followed by *Enitens* and *Macarthurii* showing that these genotypes are currently the best in this multiple response optimisation goal (Tab. 5). Picking up the darkest green genotype for a response identifies the best genotype for that response variable. For example, if more emphasis is on supplying a customer that is known to demand a product with high viscosity level, it would be ideal to process large quantities of dissolving wood pulp from *Edunnii* species, and so on. For convenience, it will be nice to have Tab. 5 as a chart display in a wood dissolving pulp plant for easy reference and educational purposes to employees.

Tab. 5: Desirability matrix of the refined overlaid contours.

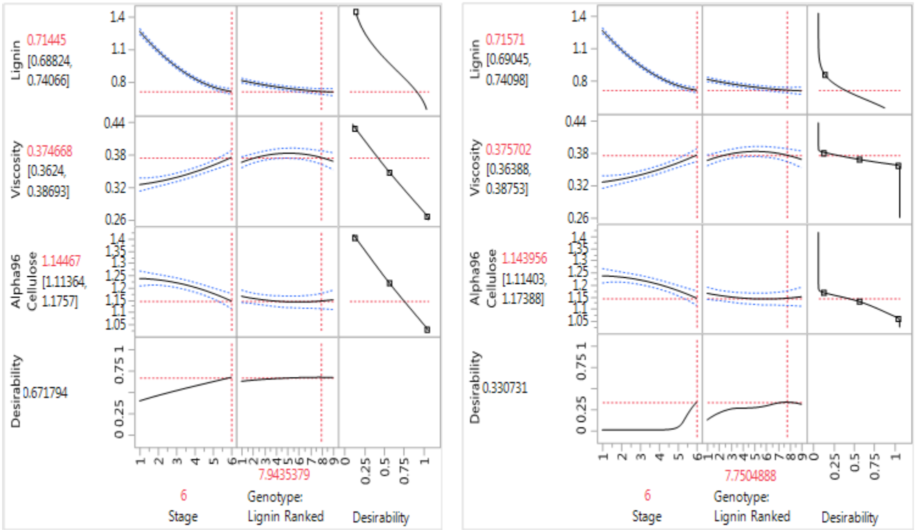
		Genotype_Lignin Ranked Resistant →								Least
Response Variables		1-Edunnii	2-Esmithii	3-GCG438	4-Egrandis	5-GUA380	6-GUW962	7-Macarthurii	8-Emearnsii	9-Enitens
Lignin	Rank	1	2	3	4	5	6	7	8	9
	Actual	0,3775	0,3334	0,2953	0,2690	0,2477	0,2293	0,2106	0,2011	0,1944
Viscosity	Rank	7	5	3	2	1	2	1	4	6
	Actual	32,0065	29,9824	28,6368	28,1204	27,6160	28,1204	28,3771	29,7068	31,4106
Alpha96 Cellulose	Rank	1	2	4	5	6	8	9	7	3
	Actual	95,0639	95,2827	95,4758	95,5313	95,5590	95,6426	95,6705	95,5869	95,4481
Overall Score		9	9	10	11	12	16	17	19	18
		Edunnii	Esmithii	GCG438	Egrandis	GUA380	GUW962	Macarthurii	Emearnsii	Enitens
Desirability level		**33%**	**33%**	**37%**	**41%**	**44%**	**59%**	**63%**	**70%**	**67%**

The desirability functions were used to project optimal solutions on permutations of different objectives. Further exploration into the experimental region of the pulping process discovered several products ingredient mix of which Fig. 5 and Fig. 6 show the chosen objectives of interest. In all the cases α-96 cellulose was first minimized (maximising the raw α-96 cellulose) followed by tailoring lignin and viscosity to achieve the set objective.

Fig. 5a: The objective was to have a process that can produce pulp based on the range of the observed variables, with medians of the responses as target, the minimum and maximum values considered as the lower and upper boundaries. The corresponding lignin, viscosity and α-96 cellulose values for the set point were 0.2017, 29.5246 cP and 95.596% respectively.

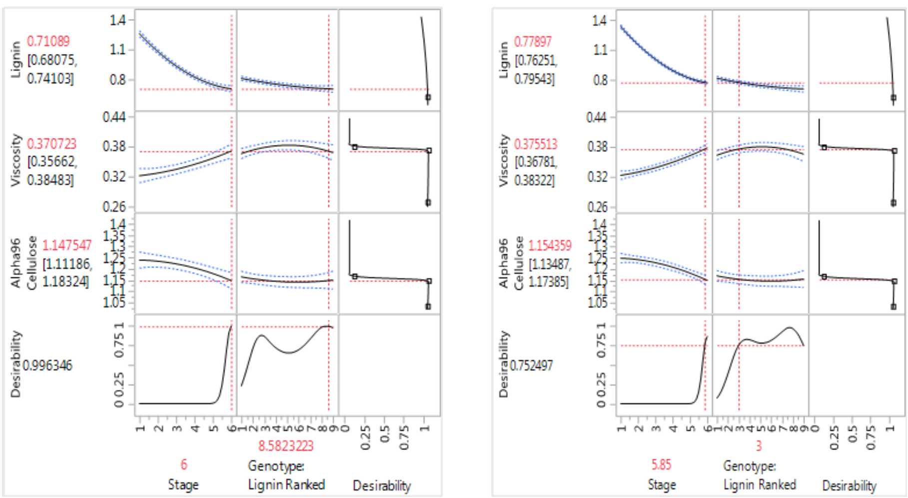
A desirability level of 0.67 was achieved which seems to be a fairly easy task for the production team.

Fig. 5b: Setting of the objective that produce output from the best qualities of all the responses lignin (0 - 0.5); viscosity (28 - 35) and α-96 cellulose (95 - 98) where the target becomes the midpoint of the desired ranges. The Genotype code 8 (*Emearnsii*) provided the best output in the final stage of processing. However, redefining the limits of the experimental region to such a high standard based on the ideal responses throughout is closer to impractical as the desirability drops drastically to 0.33. This indicates the difficulty in working within such a tight space but nevertheless *Emearnsii* could still provide the best optimal results in the final stage of wood dissolving pulp process where the results were as follows: lignin (0.2034), viscosity (29.2454 cP) and α-96 cellulose output (96.6159%).



(a) (b)

Fig. 5: Unrestrained and restrained factor setting solutions.



(a) (b)

Fig. 6: Constraints for increasing desirability and the number of genotype mix.

Fig. 6a: Setting the targets very close to what the process can achieve, raised the desirability level extensively. The desirability analysis results show that Genotype 9 (*Enitens*) is capable of easily (Desirability = 0.9963) producing these results with a much higher molecular weight (viscosity = 30.6222cP), lignin reduction going down to as low as 0.1969 and α-96 cellulose 95.5161%. There is a possibility that genotype 3(*GCG438*) may also perform better at these response constraints as depicted by the second pick of the desirability after that of Genotype 9 (Enitens)

Fig. 6b: The most ideal objective was to set constraints that are viable from a business point of view. Economically and practically processing few genotypes that can achieve the best results may not meet the pulp demand capacity. The JMP response profiler indicated that there is a possibility of mixing seven of the genotypes (*GCG438*, *Egrandis*, *GUA380*, *GUW962*, *Macarthurii*, *Emearnsii* and *Enitens)* to produce maximum α-cellulose of up to 95.3278% which is the threshold to the ideal α-96 cellulose requirements. The corresponding lignin level (0.3044) will be slightly above the target of k-number = 0.25 and viscosity (29.2961cP) well within the required range of 28-35cP. The most resistant species (*Edunnii* and *Esmithii*) to lignin reduction were dropped out from this mixture. The seven genotypes achieved a desirability level of 0.7525 suggesting that the product mix is manageable.

CONCLUSIONS

The response surface methodology (RSM) was used to model each response variable to determine the optimum region. The desirability analysis was used to consolidate the optimisation components from the RSM models. In order to develop better models that satisfy the model building assumptions, the response variables were transformed. The transformations improved model fitting, accuracy, understanding and consequently an enhanced predictive power.

The response surface approach identified *GUA380* as the best genotype for a higher proportion of α-96 cellulose, *Edunnii* for high viscosity and Macarthurii for the least amount of lignin content. All the investigated genotypes were capable of producing the optimal results (>95.55% α-cellulose) although a few were only at the thresholds of the feasible region. *GUA380* was the weakest *Eucalyptus* genotype in the feasible region due to its much lower viscosity levels. *Egrandis* and *GUW962* were also struggling with low viscosity properties behind *GUA380*. *Emearnsii* possesses the most desirable properties for the α-96 cellulose product output and should be considered as the ideal raw material. Tightening up the product specifications has seen *Emearnsiis* still standing out to be the most recommended but lowers the desirability level. It is far less achievable to run a process with more restricted specifications such as attempting to produce a product with all the quality characteristics as the desirability level suddenly plunges down to 0.33. On the other hand, setting the targets to what the process can easily achieve revealed that Emearnsii, although it is the best, cannot be that easy to work with as compared to *Enitens*. *GCG438* follows in producing the results consistently close to the target. The most resistant *Eucalyptus* species (*Edunnii* and *Esmithii*) are the most difficult to process. The other seven can possibly be mixed during processing and have a 0.75 desirability level which is economically viable than to consider few genotypes with a higher desirability level but incapable of meeting the pulp demand capacity.

The drawback to the RSM is that the surface does not precisely show the actual behavioural patterns of the responses. Further studies may require other models such as generalised additive models and fractional polynomials to deal with this situation. It would have been better if the data considered the supply capacity as well, rather than simply selecting the best genotypes, yet their availability is less than what the mills require. Plantation size and more detail on the site quality may be required for future research in order to suggest solutions that are also practically possible.

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