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Research article

Optimization of pulp production from groundnut shells using chemical pulping at low temperatures

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ABSTRACT

Paper production through chemical pulping has been identified as one of the ideal avenues of exploring the uses of groundnut shells as they are rich in cellulose. Ideally, the cellulose can be used to synthesize fibres that can be converted into useful paper products. In this study, chemical pulping was the chosen process for liberating the fibres as it is effective in dissolving lignin embedded within the cellulose. In addition, the fibres produced have superior physical properties compared to mechanical pulping. It is imperative that optimal conditions are identified for the chemical treatment process, in order to ensure that energy and chemical consumption are minimized. All these measures are aimed at reducing production costs and make chemical pulping economically viable, as compared to the mechanical pulping process which is less costly. Response surface methodology (RSM) was used in this study to evaluate the effect of three independent variables (cooking time, temperature, and sulphidity) on pulp yield and kappa number. These parameters are critical in the chemical pulping process and the optimal conditions obtained were 180 min, 100 °C and 23.6 wt.%, respectively. At the optimal cinditions, the pulp yield was 64.39wt% with a kappa number of 19.5. The results showed that all parameters investigated, had a statistically significant effect on the production of pulp. The increased cooking time was efficient in ensuring complete impregnation of the groundnut shells with chemicals for pulping and ensuring that the dissolution of lignin is not selective and does not result in dead spots inherently compromising the quality of the pulp. On the other hand, lower temperatures limited the peeling effect due to hydrolysis of carbohydrates which increased pulp yield due to a higher cellulose retention. Consequently, this contributed towards obtaining pulp that is well cooked, has a low bleach consumption and a higher quality.

1. Introduction

Current challenges in the pulp and paper making industry include attaining affordable quality pulp while preserving the environment by reducing the energy, water and chemical requirements during the pulping process (Laftah and Abdul Rahaman, 2015). The bulk of fibres for pulp production world-wide are derived from wood which has increased deforestation and poses an environmental degradation (Kamoga et al., 2013). Wood products including paper account for 10% of total deforestation, whilst deforestation is responsible for 12% of greenhouse gas emissions world-wide (Corcelli et al., 2018). To mitigate deforestation, it is imperative to investigate alternative sources of making pulp with similar characteristics to wood. Most lignocellulose-based agricultural residues have unique cellular arrangements, composition and characteristics which make them suitable for many applications such as production of composites, fuel, textiles and paper (Fuqua et al., 2012).

Amongst several agricultural wastes, groundnut shells have emanated as one of the viable agricultural residues for pulp production due to a substantial cellulose content which is the primary raw material required for pulp production (Ramgopal et al., 2016). Groundnut shells have the potential to complement conventional wood supplies as currently they are being under-utilized in countries that are heavily dependent on agriculture such as Zimbabwe (Wedin et al., 2010). In the 2018/2019 farming season in Zimbabwe, more than 200 000 ha of land was cultivated for groundnuts production, with an estimated harvest of 100 000 tons at a yield of 0.5 tons per hectare. Groundnut shells account for 20% of the dried peanut by weight, which is rich in hemicellulose, cellulose and lignin. This entails that about 20 000 tons of inexpensive groundnut

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shells are available for value addition. The Grain Marketing Board (GMB) is the main buyer of groundnuts and in Zimbabwe, it conducts shelling at industrial level at its processing plant in Ruwa. This is a great opportunity for the pulp and paper industry in Zimbabwe as it has adversely been affected by lack of cheap, readily available raw materials to counter importation (eBusiness Weekly, 2020).

The paper making process involves a number of stages including debarking, pulping, bleaching and papermaking (Lehto and Alén, 2015). Pulping is meant to reduce the lignocellulosic material in the raw material to pulp by mechanical, chemical or a mixture of both methods (Shukry et al., 2007). Although mechanical pulping has lower costs and higher yields it is not effective in removing lignin and produces pulp of a lower quality as tensile strength and elongation reduce with increase in the lignin content (Laftah and Rahman, 2016). Therefore, chemical pulping (which removes lignin) was chosen for this research as it will result in pulp of higher quality. Parameters that affect the chemical pulping process were optimized to obtain a high pulp yield at reduced energy and chemical consumption using response surface methodology.

Response surface methodology is an effective tool which uses a sequence of designed experiments to obtain an optimal response. It allows analysis of the simultaneous effects of multiple independent variables while requiring a small number of experiments. This is a favourable method for analysis of chemical and biochemical processes including chemical pulping (Berhanu et al., 2018). The analysis of variance (ANOVA) for experimental results was used to identify significant effects of the parameters under investigation as well as to check the statistical significance. Response surface methodology was used to investigate and optimize the following parameters:

1.1. Cooking temperature

The effect of temperature on the pulping of groundnut shells was evaluated with the objective of maximizing the rate of delignification and reducing the energy requirements. The temperature ranges for chemical pulping are usually in the range 140–180 °C, but also depends on the raw material being used for pulping, considerably lower temperatures were investigated in this research, a temperature range of 84–100 °C was used to reduce energy requirements (Pydimalla et al., 2019).

1.2. Sulphidity

Addition of sodium sulphide to sodium hydroxide increases the rate of delignification whereas sulphidity represents the amount of sodium sulphide added as a percentage. To ensure that the process is economically, and environmentally sustainable minimal chemical consumption is desirable.

1.3. Cooking time

Pulp cooking time is usually maintained between 90-120 min (Wan Rosli et al., 2009) depending on the characteristics of the raw materials used to derive fibre material. A higher cooking time is imperative to ensure that all the raw material is impregnated with pulping chemicals as the particles move through a complicated path along the media (Tavast and Brännvall, 2017).

1.4. Pulp yield

Pulp yield was used to assess the impact of varying the first three parameters during chemical pulping. The dry mass of pulp produced from the chemical pulping was weighed and the pulp yield was determined using Eq. (1):

$$Pulp\ yield = \frac{dry\ mass\ of\ pulp}{Oven\ dry\ mass\ of\ groundnut\ shell\ sample\ at\ the\ beginning} \times 100\% \quad (1)$$

1.5. Kappa number analysis

Kappa number indicates the amount of lignin in the pulp and shows the extent of delignification after pulping. A high kappa number indicates that lignin content is high (Berhanu et al., 2018). In this study, kappa number analysis was carried out according to ISO 302:2004. This method is applicable to all chemical and semi chemical pulps and gives a Kappa number in the range of 1–100.

The effects of the aforementioned process parameters temperature, sulphidity and cooking time on the delignification of ground nut shells with kappa number and pulp yield as performance indicators was investigated in this study.

2. Materials and methods

2.1. Groundnut shell preparation

A sample of groundnut shells was washed with distilled water for 5 min to remove any material attached to the shells. Thereafter, the groundnut shells were ground in a blender to reduce their size and increase the surface area for increased contact with the chemicals during the cooking process. The groundnut shells were then dried with air for 24 h and placed in an oven for 1 h at 100 °C for further drying.

2.2. Cooking liquor preparation

A solution of NaOH and Na_2S was prepared using distilled water by mixing the two to form a white liquor. The active alkali (NaOH + Na_2S) was 20% the weight of the groundnut shells.

2.3. Cooking process

About 50g of oven dry groundnut shells were weighed and placed in a 1000 ml beaker and 250 ml of cooking liquor was added to the beaker to ensure a solids to liquid ratio of 4:1. Heat was added to the contents of the beaker to reach required cooking temperature at the required cooking time. After cooking, the pulp was washed using distilled water to remove black liquor until a clear colour of water was maintained. Thereafter the pulp size was reduced using a blender.

2.4. Paper sheet making process

The pulp produced from the cooking process at optimal conditions was used to make paper sheets using a manual method on a deckle. The cooked pulp was screened into sizes of between 90 and 180 μm. The pulp was then poured into a large container, dispersed to distribute the pulps, collected in a deckle and water was drained out while spreading the pulp evenly on the deckle. The pulp was transferred to a cloth and compressed to drain water and ensure an even thickness to produce sheets that were then oven dried for 2 h. The procedure is summarized in Figure 1. The surface morphology of the paper sheets was analyzed using a scanning electron microscope (SEM).

2.5. Experimental design

Response surface modelling was used to deduce the optimal conditions of the three independent variables. The three independent variables investigated were temperature ($80 < T < 100$ °C); cooking time ($180 < t < 240$ min); and sulphidity ($0 < S < 30$ wt.%) on the depended variables of kappa number and pulp yield. A total of 20 experiments were performed to formulate model equations and to see interaction effects of

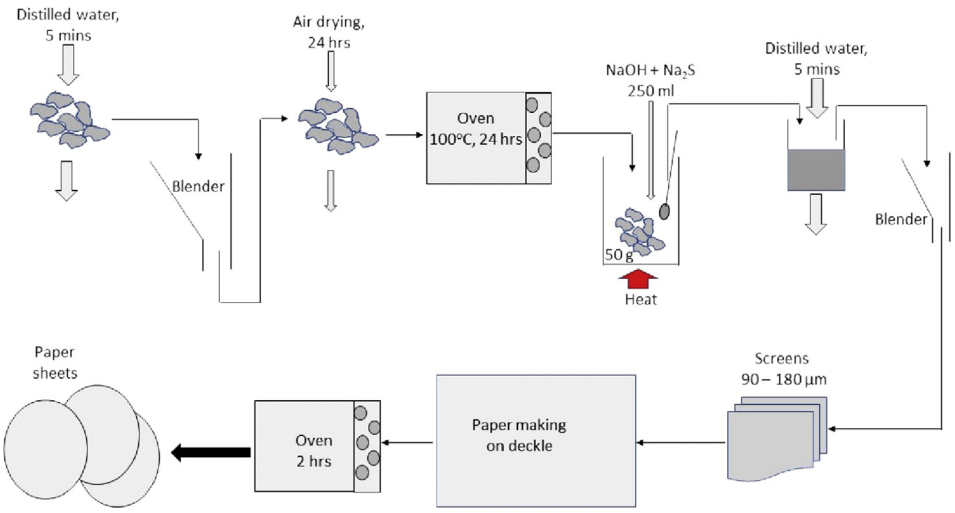

Figure 1. Process flow diagram.

each parameter. A full 2^k factorial design was used to fit a linear regression model with 6 centre points and 6 axial points. The axial points were added to allow estimation of the quadratic terms of the model in a central composite design. Analysis of variance (ANOVA) was used to test the significance of the regression model at 99% confidence interval. Central composite design was used to study the surface response for pulp yield and kappa number was carried out using a quadratic model with factors T, t, S, Tt, tS, ST, T^2, t^2, S^2 and the intercept. Analysis of the factors as functions of the model were carried out. T, t, and S represent cooking temperature, cooking time and sulphidity, respectively. The coefficients were used to study the response variables. The *p-Value* resulting from ANOVA gives the coefficient relationship with the error.

3. Results and discussion

3.1. Response surface modelling and ANOVA

Table 1 below shows the parameters that were investigated and the yield and kappa number responses.

The models predicted for pulp yield and kappa number are given by Eqs. (2) and (3) as follows:

$$R_Y = -39.0 + 2.757T - 0.0161t - 0.6131S - 0.01484T^*T - 0.000139t^*t + 0.00292S^*S - 0.001130T^*t + 0.00122T^*S + 0.001122t^*S \quad (2)$$

Table 1. Experimental design of the cooking tests and responses.

Run	Temperature (°C) (T)	Time (min) (t)	Sulphidity (%) (S)	X(T)	X(t)	X(S)	Yield (%)	Kappa Number
Factorial Points								
1	84	204	6	-1	-1	-1	69.400	27.790
2	96	204	6	1	-1	-1	67.780	25.780
3	84	276	6	-1	1	-1	66.921	25.567
4	96	276	6	1	1	-1	63.600	20.330
5	84	204	24	-1	-1	1	66.223	25.500
6	96	204	24	1	-1	1	64.120	20.950
7	84	276	24	-1	1	1	64.430	21.980
8	96	276	24	1	1	1	62.110	19.490
Axial Points								
9	80	240	15	-1.682	0	0	65.720	24.620
10	100	240	15	1.682	0	0	63.010	19.554
11	90	180	15	0	-1.682	0	68.290	25.960
12	90	300	15	0	1.682	0	64.600	24.530
13	90	240	0	0	0	-1.682	68.600	26.520
14	90	240	30	0	0	1.682	64.500	23.050
Centre Points								
15	90	240	15	0	0	0	65.800	24.775
16	90	240	15	0	0	0	65.780	24.770
17	90	240	15	0	0	0	65.795	24.760
18	90	240	15	0	0	0	65.780	24.765
19	90	240	15	0	0	0	65.770	24.770
20	90	240	15	0	0	0	65.790	24.775

t- Time (min); T- Temperature (^{o}C); S- (sulphidity-wt.%).

Table 2. ANOVA for Response Surface model for Pulp yield.

Source	Sum of Squares	Degrees of Freedom	Mean Square	*F*-Value	*p*-Value
Model	64.5657	9	7.1740	56.44	2.17E-7
Residual	1.2710	10	0.1271		
Lack of fit	1.2704	5	0.2541		2.9E-8
Pure Error	0.0006	5	0.0001		
Total	65.8367	19			

Table 3. ANOVA for response Surface model for Kappa Number.

Source	Sum of Squares	Degrees of Freedom	Mean Square	*F*-Value	*P*-Value
Model	90.7664	9	10.085	22.12	1.86E-5
Residual	4.5595	10	0.4560		
Lack of fit	4.5593	5	0.9119		7.52E-5
Pure Error	0.000171	5	0.000034		
Total	95.326	19			

$$R_K = -177.5 + 4.911T - 0.014t - 0.013S - 0.02821T^*T - 0.000013t^*t - 0.00055S^*S - 0.00023T^*t + 0.00334T^*S + 0.000753t^*S \quad (3)$$

Minitab was used to conduct regression analysis and lack of fit test and results of ANOVA are shown in Tables 2 and 3. From Table 2, for the response model of pulp yield, the p-Value for the model is very small, 2.17E-4 (<0.01), indicating a strong relationship between the process variables and pulp yield. The ANOVA shows a statistically significant relationship between the variables with a 99% confidence level.

The goodness of fit of the model for the response (R_P) for pulp yield was verified using the coefficient of determination, R^2 and absolute average deviation (AAD) which was equal to 0.9807 and 1.35. The R^2 value means that 98.07% of the variability of the response can be explained by the adjusted model. Whereas, the lower value of (AAD) indicates that predictions using the model have less variance.

From Table 3, for the response model of kappa number, the p-Value for the model is very small, 1.86E-5 (<0.01), indicating a strong relationship between the process variables and kappa number. The goodness of fit of the model for the response (R_K) was verified using the coefficient of determination, R^2, as well as the AAD which were 0.9091 and 1.87, respectively. This means that the adjusted model can be used to explain variability of the model up to 90.91%. These values entail that the model has a statistically insignificant lack of fit and guarantees that convergence at the actual optimum experimental conditions are achieved.

3.2. Pulp yield

In the central composite design presented in Table 1 the effects of three independent variables temperature (80 < T < 100 °C); cooking time (180 < t < 240 min) and sulphidity (0 < S < 30 wt.%) were evaluated on pulp yield and kappa number. The significant interaction effects on responses are demonstrated in the three-dimensional response surface plots below.

Figure 2 shows that there was a sharp decrease in pulp yield for the sulphidity and cooking time interaction as these parameters were increased. This is attributed to carbohydrate degradation through hydrolysis reactions and lignin removal (Wan Rosli et al., 2009). On the other hand, Figure 3 shows a slight increase in the yield at lower temperatures and sulphidity due to lignin being deposited on the fibres as condensation reactions are favoured at lower temperatures. As temperature and sulphidity increased, there was a decrease in the pulp yield, because at higher temperatures dissolution of lignin is enhanced. In addition, diffusion of pulping chemicals through the media is faster at

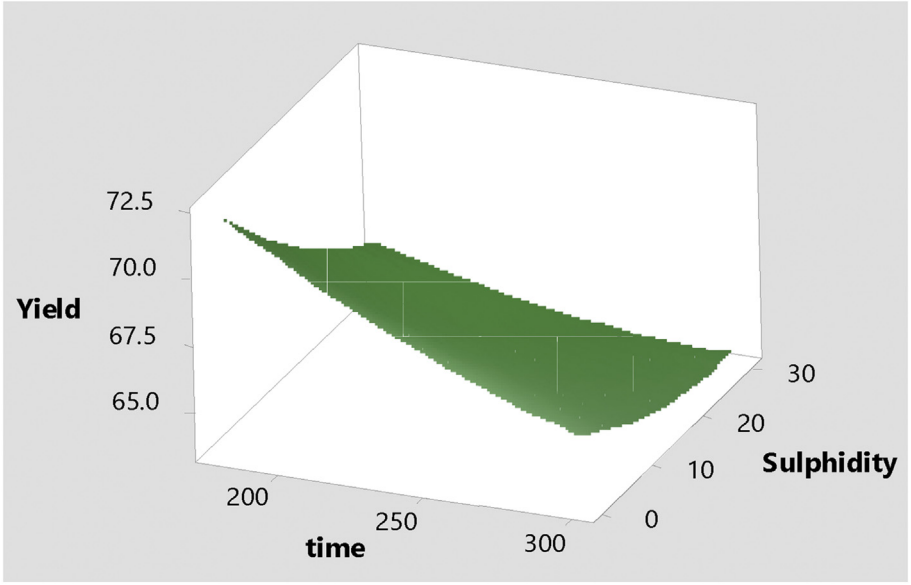

Figure 2. Surface Plot of Yield (wt.%) against sulphidity (wt.%) and time (min).

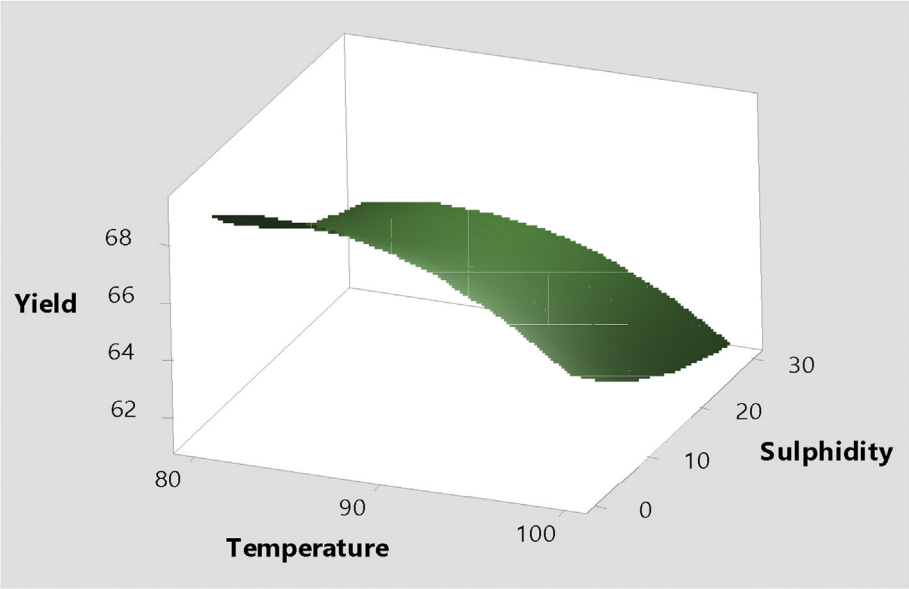

Figure 3. Plot of yield (wt.%) against temperature (^{o}C)and sulphidity (wt.%).

higher temperatures leading to a higher rate of delignification and carbohydrate hydrolysis, reducing pulp yield. Similarly Figure 4 shows a slight increase in yield at lower temperatures and thereafter a decrease. The initial increase of pulp yield at lower temperatures may be attributed to lignin inaccessibility for alkaline degradation with low diffusion rates. The reduction in pulp yield, thereafter, is due to the hydrolysis of carbohydrates of lower molecular weight which has a peeling effect on carbohydrates and inherently reduces the pulp yield (Pydimalla et al., 2019).

Figures 2, 3, and 4 in summary show that sulphidity and cooking time have the most significant impact on the pulp yield whilst temperature has moderate effects. The pulp yield obtained in this investigation was higher than that obtained by other researchers using other raw materials such as *Acacia Mangium* and *Bambusa tulda* (Wan Rosli et al., 2009). The delignification of groundnut shells in this study showed a higher pulp yield in comparison to other raw materials that have a high lignin content due to the lower intensity of the delignification process and peeling effects of carbohydrates at lower temperatures (Shukry et al., 2007).

3.3. Kappa number

The kappa numbers obtained where in the range of 19.45–27.79 which are comparable to that achieved for kraft and soda pulping for bamboo (Vu et al., 2003). Figure 5 shows the interaction effects for sulphidity and cooking time, an increase in sulphidity showed a statistically significant effect on the kappa number as compared to cooking time. Figure 6 shows that there is a considerable effect on the kappa number for the interaction between temperature and time. An increase in the concentration of pulping chemicals which is reflected by an increase in sulphidity has a desirable effect on kappa number as this improves the

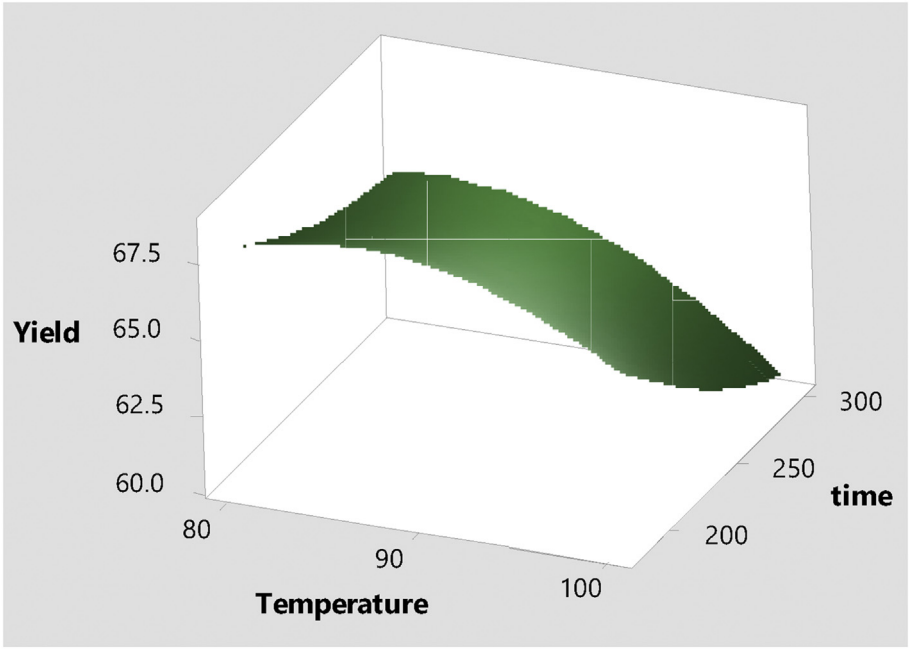

Figure 4. Surface Plot of Yield (wt.%) against temperature (^{o}C)and time (min).

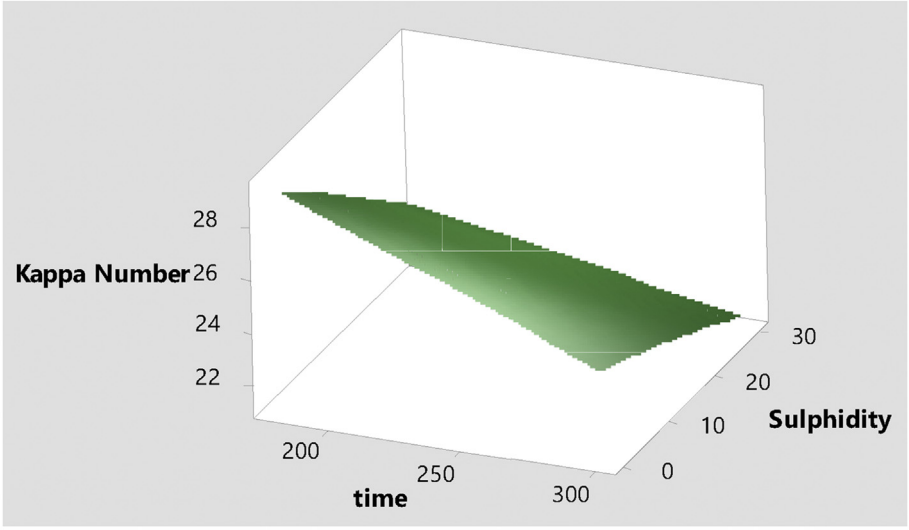

Figure 5. Kappa number against Sulphidity (wt.%) and Time (min).

lignin degradation, whilst increasing temperatures improves the diffusion of the chemicals into the material which accelerates the delignification process (Berhanu et al., 2018). Figure 7 shows a slight increase in kappa number at a lower temperature and cooking time, at these conditions the delignification process is inefficient as condensation of lignin is favoured and diffusion at low temperatures is slow. As temperature and cooking time were further increased, kappa number decreased.

3.4. Optimum conditions

Table 4 shows the optimum conditions obtained to achieve a maximum yield and the minimum kappa number.

The numerical optimal conditions for the chemical pulping were determined using design expert software by setting the desired goal for each factor and response. The optimal cooking temperature of 100 °C was obtained. However, this value is lower than the usual cooking temperatures which can be as high as 170 °C. The lower optimal temperature obtained was possible due to the prolonged cooking time which compensated for the energy requirements of the process (Wan Rosli et al., 2009). This is desirable as high cooking temperatures result in a peeling effect of the desired pulp, through hydrolysis of carbohydrates which reduces the pulp yield. In addition, chemical pulping at temperatures at 100 °C or below overcomes formation of hexenuronic acid which reacts with electrophilic bleaching chemicals increasing their consumption. Hexenuronic acid also compromises the brightness stability of pulp due to a high affinity of metal ions, which is the most important performance property for commercial pulp, as it directly impacts the quality of pulp. The optimal cooking time was found to be 180 min this was effective as cooking time less than this does not allow for effective penetration of the cooking chemicals to the middle lamella of the groundnut shells. The whole sample of the groundnut shells is required to be impregnated with pulping chemicals, for effective delignification and achieve a homogeneous cook of pulp (Vu et al., 2003). In addition, a higher cooking time also ensures that slow reacting residual phase lignin is effectively reduced (Tavast and Brännvall, 2017). Groundnut shells have a high lignin content of about 30wt% compared to other renewable sources such as rice husks which have about half the lignin content therefore a longer cooking time is required for effective lignin reduction (Ramgopal et al., 2016). Kappa number of 19.5 is relatively low and desirable as this will reduce the chemical costs for bleaching and ensure the process is more environmentally sustainable with minimal waste emissions. The kappa number can be further reduced using oxygen delignification (OD) which is an indispensable process for removal of residual lignin that remains after chemical pulping. The OD process effectively offsets downstream

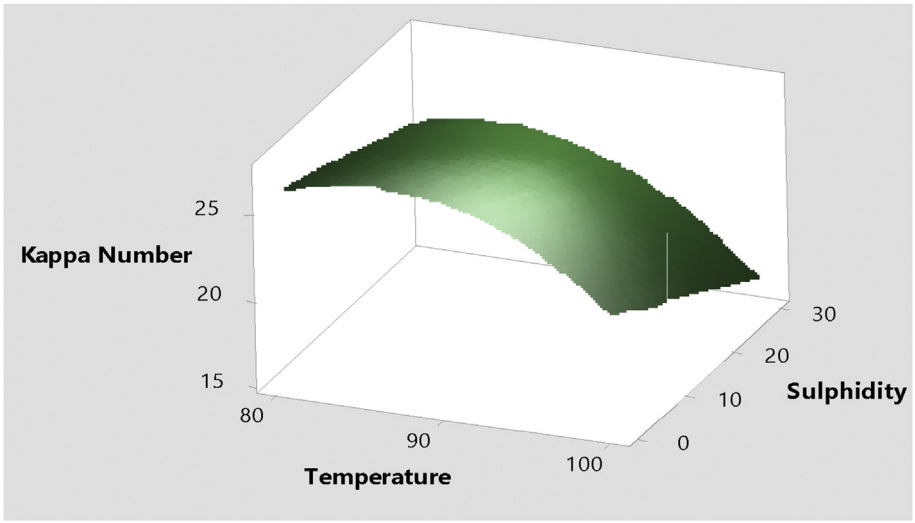

Figure 6. Kappa number against temperature (°C) and sulphidity (wt.%).

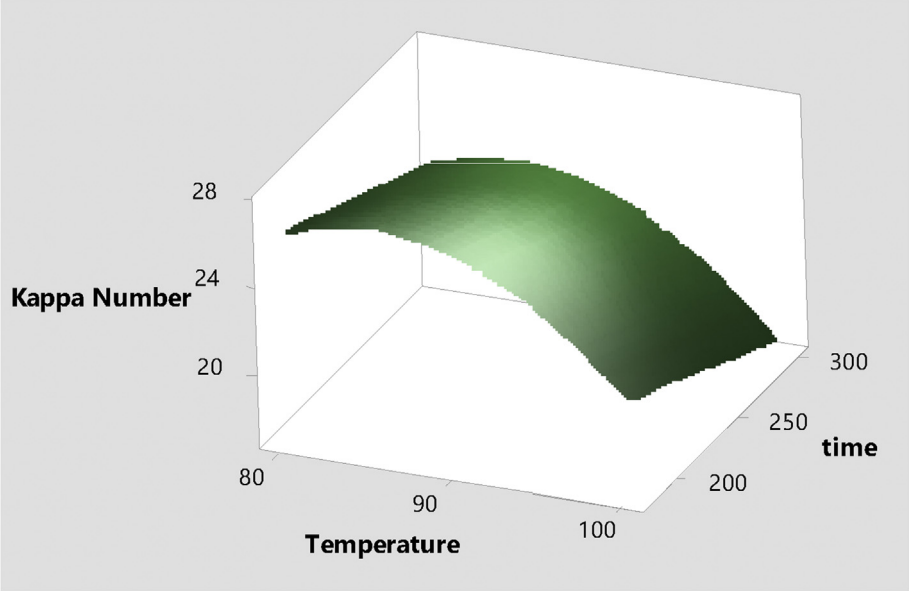

Figure 7. Kappa Number against temperature (°C) and time (min).

Table 4. Optimum conditions for chemical pulping.

Parameter	Optimum conditions
[a]Cooking Temperature (°C)	100 ± 1
[a]Cooking Time (minutes)	180.0 ± 0.5
[b]Sulphidity (%)	23.6 ± 0.1
[b]**Yield (%)**	**64.34 ± 0.13**
[b]**Kappa Number**	**19.5 ± 0.1**

[a] This was based on the sensitivity of the equipment being used.
[b] Based on calculation of the sulphidity, Yield and Kappa Number.

bleaching chemical requirements inherently reducing pollution effects on bleaching effluent. Removal of residual lignin also curtails the sources of adsorbable organic halides (AOX) which are formed when the residual lignin is oxidised (Li et al., 2016).

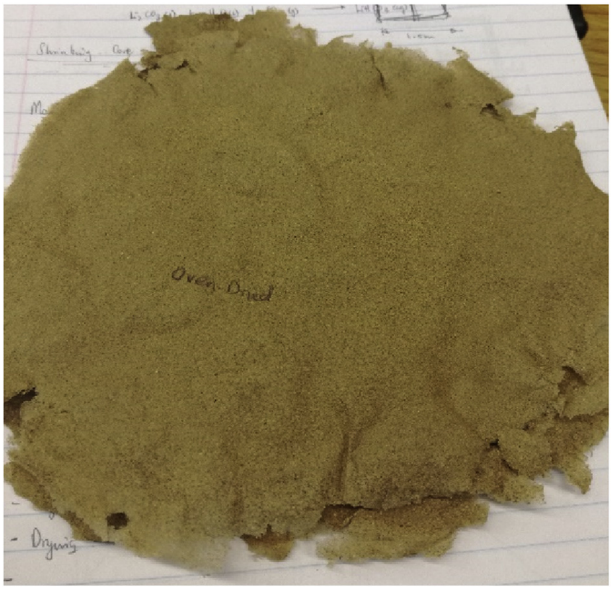

Figure 8. Oven dried sample.

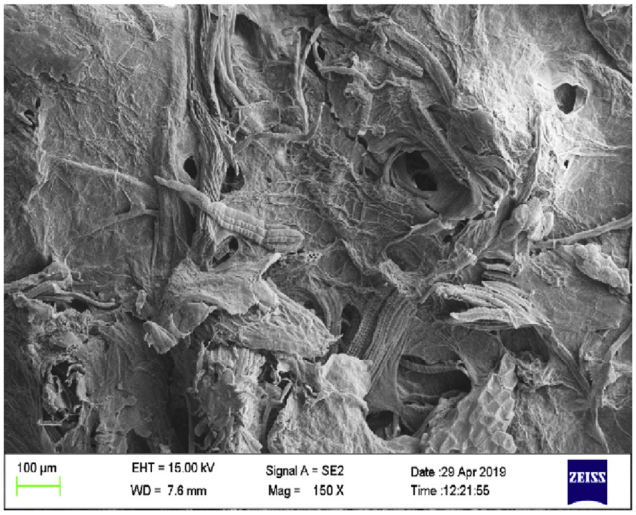

Figure 9. SEM Surface morphology at 100µm.

3.5. Synthesized oven dried paper sheet and scanning electron microscope (SEM) surface morphology

Figure 8 shows a sample of the oven dried paper produced from the pulp. It does not show visible cracks or any defects and has a consistent surface structure. In addition, it is soft and non-rigid. These results are due to the fact that the pulp was well cooked and thus produced fibres of a high quality. Figure 9 shows the morphology of the surface of the paper. The SEM image in Figure 9 shows a dense network of fibres of varying sizes entangled with micro and macro sized fibres which are cross linked thus improving the physical strength of the paper. The measured tensile strength of the paper was 3.59 MPa, this was substantially higher than that obtained by (Jones et al., 1998), this can be attributed to the lack of micro cracks as shown in Figure 8 which usually are the source of macro crack initiation and propagation. The strain to failure obtained was 0.54 which is within the typical desired range for paper samples of 0.5–1.5% (Jones et al., 1998). The average mass per square metre was measured for the oven dried paper for five of the sheets produced and was found as 98 g/m^2. The sample grammage was in the range of sack paper which has a grammage of 60–150 g/m^2 (Ek, 2009). Based on the physical properties

obtained, the paper sheets produced can be used to synthesize material used to transport cement, flour, animal feed among other products.

4. Conclusions and recommendations

Chemical pulping at low temperatures and higher cooking time is effective in reducing lignin content in groundnut shells to desirable levels that ensure fibres of a high tensile strength and elongation are produced. The surface plots showed that temperature and sulphidity have the most statistically significant effect towards delignification. Pulp yield and kappa number showed significant decrease when these two parameters were increased. The optimal chemical pulping conditions obtained for maximum pulp yield and minimum kappa number were 180 min, 100 °C and 23.6 wt.%, respectively. At these conditions, the pulp yield was 64.39wt% with a kappa number of 19.5. The physical properties of the paper sheets investigated showed that it can be applied commercially for producing packaging material for the cement, flour and animal feed industries. To further improve the use of groundnut shells in pulp production, the use of blends of groundnut shell pulp with other non-wood raw materials, recycled paper and wood should be investigated.

Declarations

Author contribution statement

P Musekiwa: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

L.B Moyo & N Hlabangana: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

T.A Mamvura, G Danha & G.S Simate: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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