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Effect of Sodium Carbonate Pretreatment and Pretreatment Duration on Total Solids of Maize Stalk: Response Surface Methodology Approach

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ABSTRACT

Maize stalks (MS) are among the most abundant lignocellulosic crop residues in Kenya, offering considerable but under-utilized energy potential. Although cellulose in such biomass releases about 15 kJ g^{-1} when burned, converting it to methane can raise the energy yield to roughly 50 kJ g^{-1}. The chief obstacle to microbial access is lignin, which shields the cellulose and hemicellulose fractions in municipal solid waste. To generate biogas efficiently from lignocellulosic feedstocks, co-digestion is often preceded by enzymatic hydrolysis, and the success of that step hinges on an effective pretreatment that opens the biomass structure. This study evaluated sodium carbonate (Na_2CO_3) as a pretreatment agent for MS, focusing on its effect on total solids (TS) and on identifying an optimum pretreatment time. A central composite design (CCD) guided the experimental matrix, combining several Na_2CO_3 concentrations with different exposure periods: 7 % for 8 days, 3 % for 4 days, 7 % for 4 days, 5 % for 6 days, 5 % for 3 days, 2 % for 6 days, 5 % for 6 days, 5 % for 6 days, 7.8 % for 6 days, 5 % for 8.8 days, 3 % for 8 days and 5 % for 6 days. Changes in TS were used as a proxy for improved digestibility. The optimal pretreatment—7 % Na_2CO_3 applied for 4 days—achieved the highest TS value, 15.15 %, corresponding to a 7.26 % increase relative to untreated MS. Statistical analysis confirmed the fitted TS response model was significant at $P < 0.05$.

Keywords: Maize stalk, Pretreatment, sodium carbonate, CCD

INTRODUCTION

Biomass resources are globally abundant as residual waste and agricultural biomass. The primary and plentiful renewable biomass resources comprise crop leftovers, including maize straw, wheat straw, and rice straw. The economy of Kenya relies on agriculture, which possesses significant potential to increase biogas production from agricultural waste and agro-industrial byproducts, including maize residues. Kenya generates around 14 million tonnes of agricultural residues each year. Maize residues are extensively produced in Kenya, encompassing around 1,600,000 hectares of arable land(Kimutai et al., 2014). The majority of them are incinerated or disposed of, leading to significant environmental issues. The use of maize residues to improve anaerobic digestion for biogas production holds considerable potential in Kenya, as maize is a predominant crop in the nation.

Numerous initiatives have been implemented to enhance the efficiency of biogas generation by employing lignocellulosic materials as the principal resource. These initiatives involve utilizing alternative pretreatment techniques and co-digestion with nutrient-dense materials. Corn straw comprises non-consumable plant material known as lignocellulose, mostly consisting of cellulose, hemicellulose, and lignin(Jørgensen et al., 2007; Khaire et al., 2021). Hemicellulose constitutes the matrix enveloping the cellulose framework, whilst lignin acts as an encrusting substance and provides a protective coating. All three components possess covalent cross-linkages between the polysaccharides and lignin, so rendering biomass a composite substance(Binder & Raines, 2010).

Pretreatment is a crucial method for cellulose conversion processes, necessary for altering the structure of cellulosic biomass to enhance the accessibility of cellulose to enzymes that convert carbohydrate polymers into fermentable sugars (Mosier et al., 2005).

The pretreatment phase is identified as the technological constraint for anaerobic digestion bioprocesses. During the pretreatment process, the compact structure of lignocellulosic material is broken, exposing cellulose fibers and thereby enhancing the quantity of material available for microbial digestion during anaerobic digestion. Pretreatment of lignocellulosic material is performed to mitigate recalcitrance by inducing chemical and structural modifications to lignin and carbohydrates(Singh et al., 2015).Prior research has documented many pretreatment approaches, including biological, chemical, thermal processes, and their combinations, to enhance substrate hydrolysis. (Anu et al., 2020). Nevertheless, a study indicates that these pretreatment procedures are costly and necessitate greater expertise and proficiency during the pretreatment process. (Mankar et al., 2021), In comparison to sodium carbonate, which is relatively affordable and readily available, it is a cost-effective solution. Sodium carbonate is comparatively less detrimental to the environment.

Maize stalks (MS), an abundant lignocellulosic residue in Kenya, hold significant potential for bioenergy generation but are limited by their high lignin content, which impedes microbial degradation and biogas production. While various pretreatment methods exist, there is limited research on the use of sodium carbonate to enhance the digestibility of MS, particularly concerning the combined effects of concentration and duration on total solids (TS). This study addresses that gap by evaluating the impact of sodium carbonate pretreatment on TS content, using a central composite design (CCD) and response surface methodology (RSM) to model and optimize the process. The findings aim to improve the utilization of MS for sustainable energy generation and environmental pollution reduction.

MATERIALS AND METHODS

Pretreatment of Maize Stalk Residues

Physical pretreatment

Samples of maize residue were obtained from the Mosoriot region in Nandi County. The maize stalks were thoroughly air-dried to reduce their moisture content (Hassan et al., 2023). To accomplish this, they were sparsely distributed and subjected to sunlight. Effective drying facilitated superior storage and inhibited the proliferation of mold or fungi.

The dimensions of maize particles were diminished to increase the available surface area for microbial activity (Saylor et al., 2020). This was accomplished by grinding the maize stalk into tiny fragments of 1-3 cm utilizing a chaff cutter(Zhao et al., 2013). They were then further pounded using a grinding mill to reduce the particle size to increase the surface area as shown in Figure 1 and packed in a dry polythene bag and stored at room temperature ready for chemical pretreatment.

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Figure 1 :Pounded maize stalk residues.

Chemical pretreatment

MS was pretreated using Na_2CO_3) and its effect on TS was studied. The MS was soaked in Na_2CO_3 solution with different percentage concentration and duration based on the design of experiment (DOE) as shown in Table 2-1. The DOE had four replicates with center points. Ten grams of MS was soaked in 200mL Na_2CO_3 solution as per DOE in Table 1. After the desired period of soaking, the chemical solution was decanted off and MS was washed with tap water until the washings were clean, colorless and neutral in pH, (Kaur & Phutela, 2016). They were then dried in the sun for 6hrs. After they dry, MS was stored in polythene bags and was used for proximate analysis (total solids). A control (untreated MS) was simultaneously analyzed in order to obtain its TS.

Experimental Design and Optimization

The Design Expert 13 software, which includes Central Composite Design (CCD), Analysis of Variance (ANOVA), and Response Surface Methodology (RSM), was utilized for optimization. The CCD was employed to ascertain the extent of variable inputs and to determine the optimal number of runs(Ghelich et al., 2019; Manmai et al., 2020). ANOVA facilitated the analysis of the regression coefficients and prediction equations, illustrating the interactions among variables. RSM was utilized to investigate the relationship between variables and the response, as well as to estimate the optimal surface area of the response's optimal values.

The total solids served as the experimental response. Model parameters were evaluated utilizing P-values.

Table 1: Design of experiment for maize stalk residue pretreatment

Run	Na_2CO_3 Concentration (%)	Duration (Days)
1	7.00	8.00.
2	3.00	4.00
3	7.00	4.00
4	5.00	6.00
5	5.00	3.17
6	2.17	6.00
7	5.00	6.00
8	5.00	6.00
9	7.82	6.00
10	5.00	8.82
11	3.00	8.00
12	5.00	6.00
Untreated MS	-	-

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Figure 2: Masses of Na_2CO_3 and maize residues while taking weight

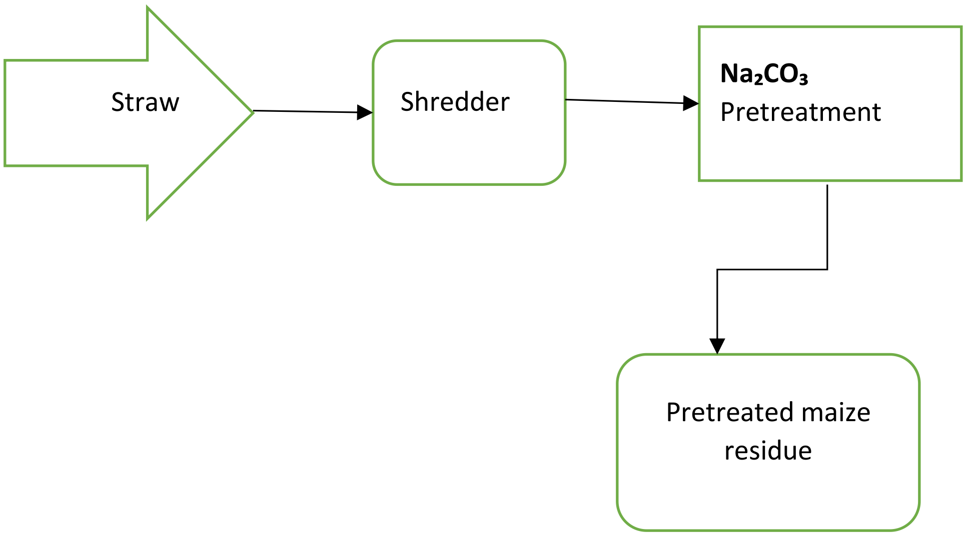

Figure 3: Pretreatment of maize stalks.

Proximate Analysis

The proximate analysis was performed in this experiment on Cow dung and maize residues based on several standards to determine the total suspended solids.

Determination of Total solids for maize stalk residue

The process of determining the moisture content of the samples involved several steps. First, the weight of an empty, dry crucible with its cover was measured using a mechanical balance. Next, 10 g of each pretreated maize stalk residue sample was placed in the crucible, and the combined weight was recorded. Each crucible, without its cover, containing the pretreated maize residue was labeled according to the corresponding sodium carbonate concentration and pretreatment duration. The labeled crucibles were then placed in an oven set at 105 °C and maintained at this temperature for 24 hours. After drying, the crucibles were removed from the oven, and the covers were immediately placed on top to prevent moisture absorption. Once the crucibles cooled to room temperature, the final weight was recorded. The total solids (TS) content was then calculated using the following equation (Wojcieszak *et al.*, 2020):

$$TS(\%) = \left[\frac{(C-A)}{(B-A)}\right] 100$$

Where:

TS = Total solids

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A = mass of dry beaker

B = mass of the dry beaker and pretreated maize residue

C = mass of dry beaker and oven maize residue (at 105°C to constant weight)

RESULTS AND DISCUSSION

Effect of Chemical Pretreatment and Pretreatment Duration

The pretreatment that gave the highest t TS was the one pretreated with 7% Na_2CO_3 concentration for four days, which gave a TS value of 15.15%, indicating an increase of 7.26% TS as shown in Table 2. It was established that the optimal conditions were 7% Na_2CO_3 (w/w) for four days, and 15.1515% TS was achieved and was subsequently used to carry out co-digestion with the core substrate, which was cow dung. alkali pretreatment hydrolyzed most organic materials and eased the anaerobic digestion process.

Table 2 : % TS for the pretreated and untreated MS.

Run	Na_2CO_3 Concentration %	Duration of Pretreatment Days	Total Solids %
1	7	8	9.54
2	3	4	11.48
3	7	4	15.15
4	5	6	7.79
5	5	3.2	12.71
6	2.17	6	6.08
7	5	6	8.02
8	5	6	8.06
9	7.82	6	10.43
10	5	8.8	8.37
11	3	8	11.61
12	5	6	8.01
Untreated MS	-	-	7.89

ANOVA for Quadratic model

Table 3 : ANOVA for Quadratic Model

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	82.07	5	16.41	6.29	0.0223	significant
A-Na_2CO_3 Concentration	7.54	1	7.54	2.89	0.1402	
B-Duration of Pretreatment	16.87	1	16.87	6.46	0.0440	
AB	8.18	1	8.18	3.13	0.1271	
A^2	13.78	1	13.78	5.28	0.0613	
B^2	43.51	1	43.51	16.67	0.0065	
Residual	15.67	6	2.61			
Lack of Fit	15.67	3	5.22			
Pure Error	0.0000	3	0.0000			

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Correctional Total	97.73	11				

The Model F-value of 6.29, as presented in Table 3, indicates that the model is statistically significant. There is only a 2.23% probability that such a high F-value could result from random variation or noise. According to Wojcieszak et al. (2020), p-values below 0.0500 suggest that the corresponding model terms are statistically significant. In this analysis, the terms B and B^2 were found to be significant. Conversely, model terms with p-values above 0.1000 are considered not significant. When a model contains several non-significant terms (excluding those needed to preserve model hierarchy), simplifying the model by removing these terms can enhance its overall performance.

The F-value of 6.29 reported in Table 3 suggests that the model is statistically significant, with only a 2.23% likelihood that such a result could arise from random variation or background noise. As noted by Wojcieszak *et al.* (2020), p-values below 0.0500 denote statistically significant model terms. In this study, the terms B and B^2 were identified as significant. On the other hand, p-values exceeding 0.1000 indicate that the associated terms are not statistically significant. When multiple non-significant terms are present—excluding those necessary for maintaining model hierarchy simplifying the model by removing them may enhance its accuracy and interpretability.

Fit Statistics

Table 4 : Fit Statistics

Std. Dev.	1.62	**R^2**	0.8397
Mean	9.32	**Adjusted R^2**	0.7062
C.V. %	17.34	**Predicted R^2**	-0.1398
		Adeq Precision	6.9388

A negative Predicted R^2 suggests that the model may perform worse than simply using the overall mean to predict the response. In such situations, a more complex model might offer improved predictive capability. Adequate Precision assesses the signal-to-noise ratio, where a value above 4 is considered acceptable. In this case, the obtained ratio of 6.939 demonstrates a sufficient signal, indicating that the model is reliable for exploring and interpreting the design space.

Final Equation in Terms of Coded Factors

$$R = +6.60 + 0.09707A - 1.45B - 1.43AB + 1.47A^2 + 2.61B^2$$

The equation expressed using coded factors allows for the prediction of the response based on specific levels of each input variable. Typically, the high levels of the factors are represented as +1, while the low levels are denoted as -1. This coded format is particularly helpful for assessing the relative influence of each factor by comparing the magnitude of their respective coefficients.

Final Equation in Terms of Actual Factors

$$R1 = +30.44414 - 1.03838A - 6.76111B - 0.357500AB + 0.366875A^2 + 0.651875B^2$$

The equation formulated using actual factor values enables prediction of the response based on specific, real-world units of each factor. In this case, the input levels must be provided in their original measurement units. However, this version of the equation is not suitable for comparing the relative influence of the factors, as the coefficients are adjusted according to the units of measurement, and the intercept does not correspond to the center point of the design space.

Diagnostics Plots

Figure 4 displays the plot comparing predicted total solids with the experimental values. The close alignment between the two, indicated by an R^2 value of 0.8397, demonstrates that the model provides a strong fit. This suggests the model is both reliable and statistically significant, making it suitable for accurately reproducing the experimental results within the studied range.

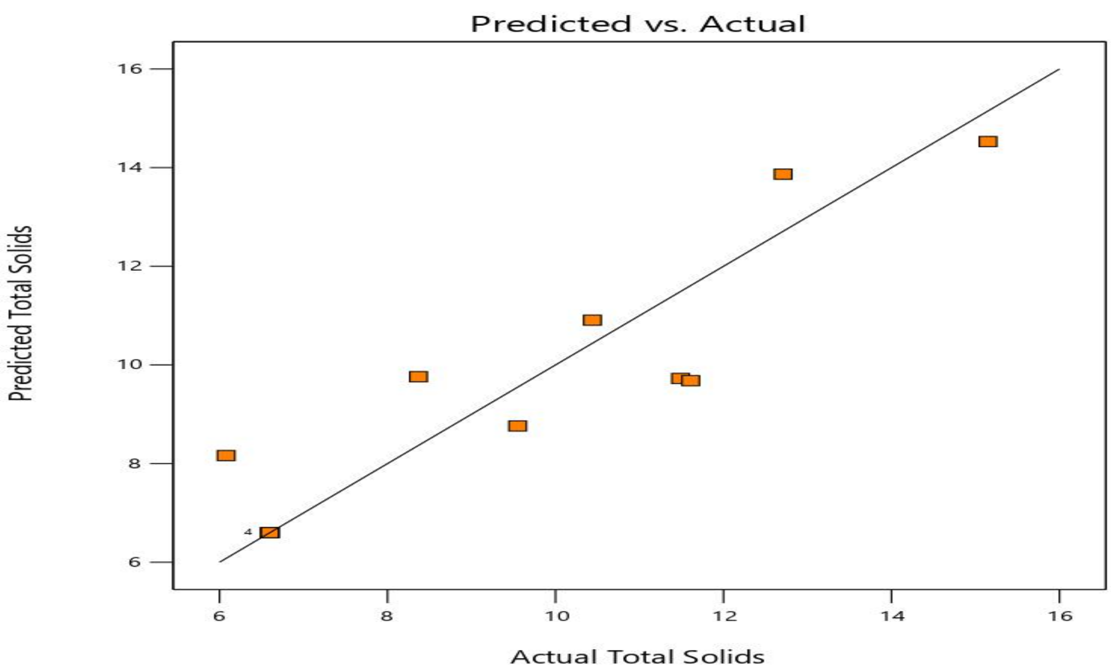

Figure 4: Plot of predicted response vs. actual Total solids.

Figure 5 illustrates the plot of externally studentized residuals versus run number, where the random scatter of residuals around the baseline indicates that the reduced cubic model is appropriate and well-fitted. In Figure 6, the normal probability plot of the externally studentized residuals is shown. The data points closely follow a straight line, suggesting that the residuals are normally distributed and that there are no apparent patterns or deviations, further supporting the validity of the model.

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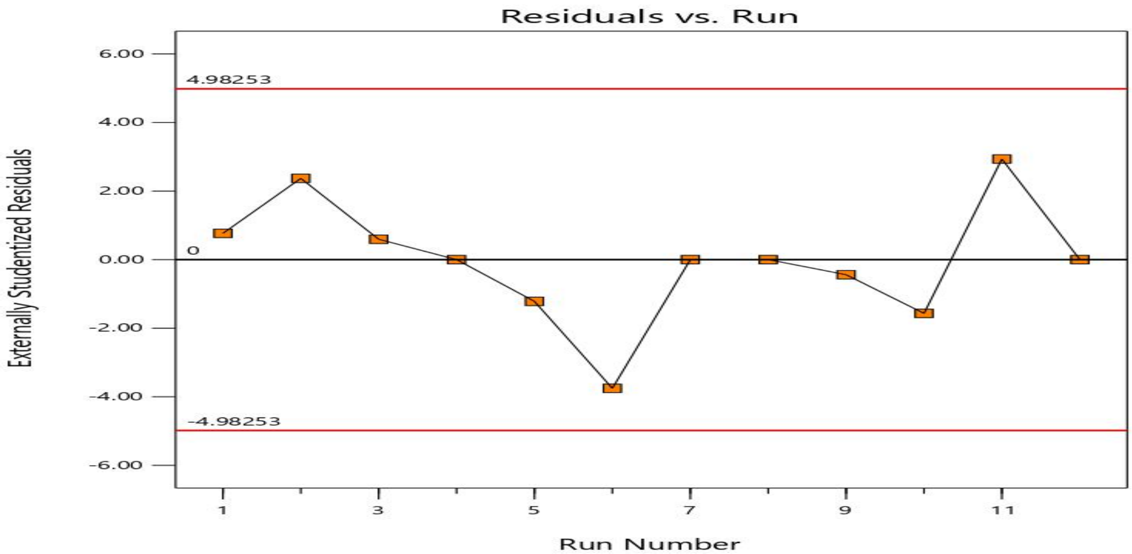

Figure 5: the plot of externally studentized residuals against the run number

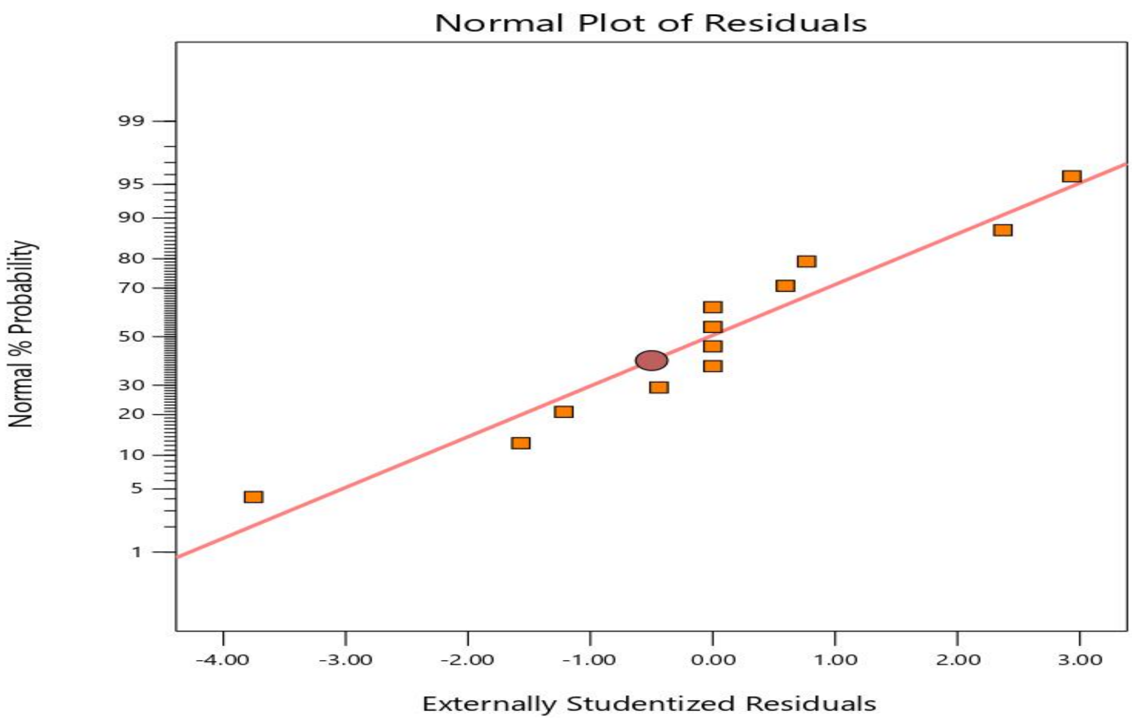

Figure 6: Normal probability plot of the externally studentized residuals

Interaction Graphs

Figures 7 and 8 illustrate the interaction between sodium carbonate (Na_2CO_3) concentration and pretreatment duration on total solids (TS). The findings indicate that this interaction significantly influences TS ($P < 0.05$), as confirmed in Table 3. Higher TS values were observed under optimal conditions, with the best results achieved at a Na_2CO_3 concentration of 7% and a pretreatment duration of 4 days.

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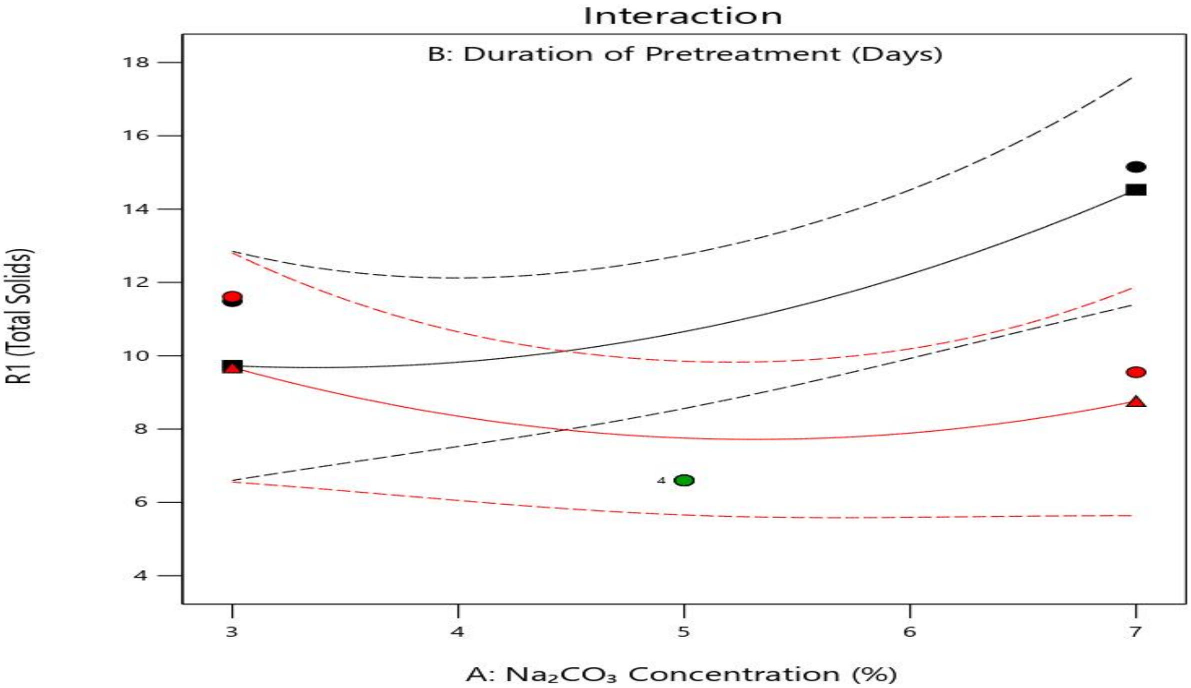

Figure 7: Interaction of Na_2CO_3 concentration and duration of pretreatment on TS.

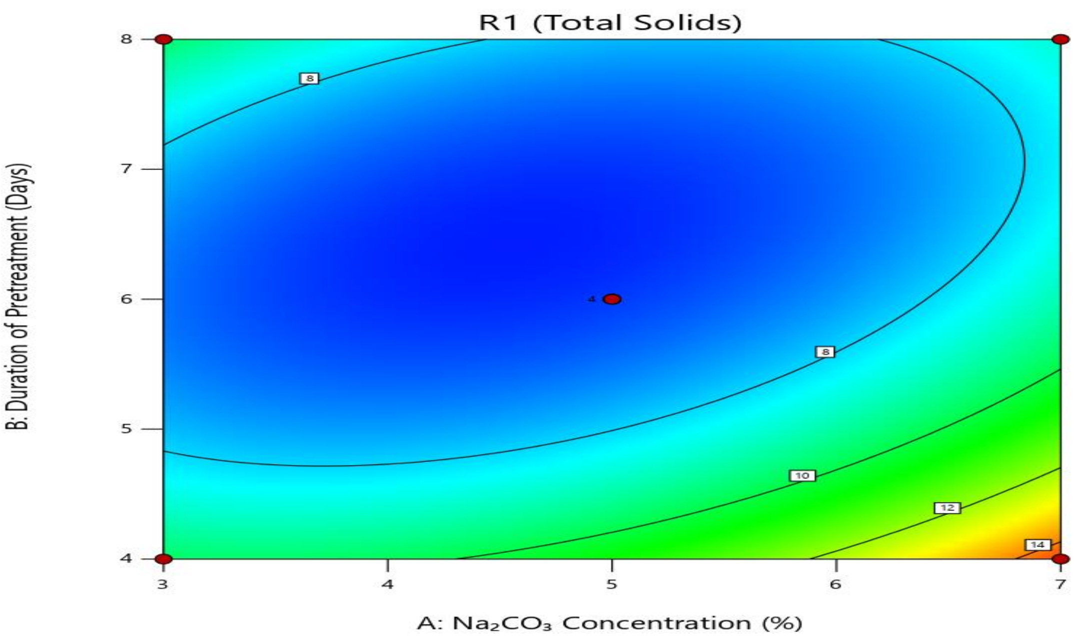

Figure 8: Effect of Na_2CO_3 concentration and pretreatment duration on Total Solids

CONCLUSION

The highest total solids (15.15 %) were attained by pretreating maize stalks with 7 % sodium carbonate for four days, a 7.26 % improvement over untreated material, which contained 7.89 % total solids. Total solids were successfully modeled as a function of the operating variables, and the model was statistically significant ($P < 0.05$). Both the sodium carbonate concentration and the length of pretreatment had a marked effect on total solids, confirming that a four-day treatment with sodium carbonate is an efficient, affordable, readily available, and comparatively eco-friendly option for enhancing maize stalk digestibility.

REFERENCES

Anu, K., Kumar, A., Rapoport, A., Kunze, G., Kumar, S., Singh, D., & Singh, B. (2020). Multifarious pretreatment strategies for lignocellulosic substrates for the generation of renewable and sustainable biofuels: A review. Renewable Energy, 160, 1228–1252. https://doi.org/10.1016/j.renene.2020.07.031

Binder, J. B., & Raines, R. T. (2010). Fermentable sugars by chemical hydrolysis of biomass. Proceedings of the National Academy of Sciences, 107(10), 4516–4521. https://doi.org/10.1073/pnas.0912073107

Ghelich, R., Jahannama, M. R., Abdizadeh, H., Torknik, F. S., & Vaezi, M. R. (2019). Central composite design (CCD)–response surface methodology (RSM) of effective electrospinning parameters on PVP-B-Hf hybrid nanofibrous composites for synthesis of HfB_2-based composite nanofibers. Composites Part B: Engineering, 166, 527–541. https://doi.org/10.1016/j.compositesb.2019.01.094

Hassan, A. A., Hasanin, M. S., & Ismail, S. A. (2023). Enzymatic valorization of cellulosic and hemicellulosic-based biomasses via the production of antioxidant water-soluble hydrolyzate of maize stalks and the green bio-deinking of mixed office waste paper. Biomass Conversion and Biorefinery. Advance online publication. https://doi.org/10.1007/s13399-023-04798-2

Jørgensen, C., Enberg, K., Dunlop, E. S., Arlinghaus, R., Boukal, D. S., Brander, K., Ernande, B., Gårdmark, A., Johnston, F., Matsumura, S., Pardoe, H., Raab, K., Silva, A., Vainikka, A., Dieckmann, U., Heino, M., & Rijnsdorp, A. D. (2007). Managing evolving fish stocks. Science, 318(5854), 1247–1248. https://doi.org/10.1126/science.1148089

Kaur, K., & Phutela, U. G. (2016). Sodium carbonate pretreatment: An approach towards desilication of paddy straw and enhancement in biogas production. Paddy and Water Environment, 14(1), 113–121. https://doi.org/10.1007/s10333-015-0483-1

Khaire, K. C., Moholkar, V. S., & Goyal, A. (2021). Bioconversion of sugarcane tops to bioethanol and other value-added products: An overview. Materials Science for Energy Technologies, 4, 54–68. https://doi.org/10.1016/j.mset.2020.12.004

Kimutai, S. K., Muumbo, A. M., Siagi, Z. O., & Kiprop, A. K. (2014). A study on agricultural residues as a substitute to firewood in Kenya: A review on major crops. International Journal of Energy Engineering, 4(9). https://www.iiste.org/

Mankar, A. R., Pandey, A., Modak, A., & Pant, K. K. (2021). Pretreatment of lignocellulosic biomass: A review on recent advances. Bioresource Technology, 334, Article 125235. https://doi.org/10.1016/j.biortech.2021.125235

Manmai, N., Unpaprom, Y., Ponnusamy, V. K., & Ramaraj, R. (2020). Bioethanol

Bima Journal of Science and Technology, Vol. 9(2B) Jul, 2025 ISSN: 2536-6041
E-ISSN: 3115-4662 DOI: 10.64290/bima.v9i2B.1290

production from the comparison between optimization of sorghum stalk and sugarcane leaf for sugar production by chemical pretreatment and enzymatic degradation. Fuel, 278, Article 118262. https://doi.org/10.1016/j.fuel.2020.118262

Mosier, N., Wyman, C., Dale, B., Elander, R., Lee, Y. Y., Holtzapple, M., & Ladisch, M. (2005). Features of promising technologies for pretreatment of lignocellulosic biomass. Bioresource Technology, 96(6), 673–685. https://doi.org/10.1016/j.biortech.2004.06.025

Saylor, B. A., Casale, F., Sultana, H., & Ferraretto, L. F. (2020). Effect of microbial inoculation and particle size on fermentation profile, aerobic stability, and ruminal in situ starch degradation of high-moisture corn ensiled for a short period. Journal of Dairy Science, 103(1), 379–395. https://doi.org/10.3168/jds.2019-16831

Singh, R. L., Singh, P. K., & Singh, R. P. (2015). Enzymatic decolorization and degradation of azo dyes: A review. International Biodeterioration & Biodegradation, 104, 21–31. https://doi.org/10.1016/j.ibiod.2015.04.027

Wojcieszak, D., Przybył, J., Ratajczak, I., Goliński, P., Janczak, D., Waśkiewicz, A., Szentner, K., & Woźniak, M. (2020). Chemical composition of maize stover fraction versus methane yield and energy value in the fermentation process. Energy, 198, Article 117258. https://doi.org/10.1016/j.energy.2020.117258

Zhao, X., Chen, J., Chen, F., Wang, X., Zhu, Q., & Ao, Q. (2013). Surface characterization of corn stalk superfine powder studied by FTIR and XRD. Colloids and Surfaces B: Biointerfaces, 104, 207–213. https://doi.org/10.1016/j.colsurfb.2012.12.003.