

Research Application Summary

Processing of leafy plant fibres in Swaziland for multiple end uses

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Abstract

Natural resources have a potential to contribute to national economies, particularly in developing countries. The handcraft sector in Swaziland relies on sisal fibres, which however, are restrictive due to their poor wearable properties. This study explored the use of enzymes as an alternative method of surface modification of sisal, century plant, and pineapple fibres for improved physical properties including dyeability and pliability. The participatory rural appraisal (PRA) phase, aimed at getting an insight into the fibre processing, was done first. Subsequently, Miller (1959)'s Dinitrosalicylic (DNS) method was used for monitoring hydrolysis and optimizing the enzymes. Conventional alkali treatment of NaOH was used as a reference. Treated and untreated fibres were dyed with Astrazon Yellow 8 GL commercial dye for 1 hour at 60°C. Obtained dye solutions were analyzed using a Shimadzu UV-VIS Spectrophotometer. Chemical and mechanical properties of raw and treated fibres were analyzed using the Fourier Transform Infra-Red (FTIR) spectroscopy, Scanning Electron Microscopy (SEM) and Instron tensile tester. Findings from the PRA revealed that fibre processors were only using sisal fibres and the production was mostly done by females (95%), some of whom were family providers. The alkaline treatment reduced sisal tensile strength by 32% while enzyme reduced it by 19%. Scanning electron microscope micrograms showed smoother enzyme-treated fibres than NaOH-treated ones. Enzyme treated fibres improved dyeability compared to raw fibres and the enzymes were effective in softening fibres and not a threat on the environment as opposed to the alkali treatment currently being used. Recovery mechanisms of enzymes for reuse could be explored and it could offset their initial high purchase cost. There is a potential in developing new products using treated fibres.

Key words: Decortication, dyeability, environmental impact, enzymatic hydrolysis, hemicellulase, lignin peroxidase, pectinase, lignocellulosics, pliability, sisal fibres

Résumé

Les ressources naturelles ont un potentiel de contribuer aux économies nationales, en particulier dans les pays en développement. Le secteur de l'artisanat au Swaziland dépend des fibres de sisal, qui, cependant, sont restrictives en raison de leurs propriétés habillables pauvres. Cette étude a exploré l'utilisation d'enzymes comme une méthode alternative de modification de surface des fibres de sisal, plante sèche, et ananas pour les propriétés physiques améliorées, y compris aptitude à la teinture et la souplesse. Premièrement, la phase de l'évaluation participative rurale (EPR) visant à obtenir un aperçu du traitement des fibres, a été faite. Ensuite, la méthode dinitrosalicylique (DNS) de Miller (1959) a été utilisée pour la surveillance de l'hydrolyse et l'optimisation des enzymes. Un traitement alcalin classique au NaOH a été utilisé comme référence. Les fibres traitées et non traitées ont été teintées avec un colorant commercial *Astrazon Yellow 8 GL* pendant 1 heure à 60°C. Les solutions de colorant obtenues ont été analysées en utilisant un Shimadzu spectrophotomètre UV-VIS. Les propriétés chimiques et mécaniques des fibres brutes et traitées ont été analysées en utilisant le Fourier Transform à infrarouge (FTIR), la microscopie électronique à balayage (SEM) et le testeur de traction Instron. Les résultats de l'EPR ont révélé que des transformateurs de fibres utilisaient seulement des fibres de sisal et que la production était principalement réalisée par les femmes (95%), dont certaines étaient des pourvoyeuses de leurs familles. Le traitement alcalin a réduit la résistance à la traction du sisal de 32%, tandis que l'enzyme l'a réduite de 19%. Les microgrammes du scanning par microscopie électronique ont montré des fibres traitées avec des enzymes plus lisses que celles traitées par du NaOH. Les fibres traitées par enzymes ont amélioré la facilité de teinture par rapport aux fibres brutes et les enzymes se sont prouvées efficaces pour le ramollissement des fibres et sans être une menace pour l'environnement, contrairement au traitement alcalin couramment utilisé. Les mécanismes de récupération des enzymes pour la réutilisation pourraient être explorés et il pourrait compenser leur prix d'achat initial élevé. Il y a un potentiel dans le développement de nouveaux produits en utilisant des fibres traitées.

Mots clés: la décortication, l'aptitude à la teinture, l'impact environnemental, l'hydrolyse enzymatique, l'hémicellulase, la lignine peroxydase, la pectinase, les lignocellulosiques, malléabilité, fibres de sisal

Background

Challenges on the ecosystem following the two industrial revolutions have rekindled the interest in green fibres in developing and developed countries (Kozłowski, 2000). Developed countries are today concerned with mitigating the effects of environmental pollution brought about by industrialisation. On the other hand, developing countries are mostly concerned with value addition of local natural resources for economic gains with little consideration on negative impact of the environment. In Swaziland, the handicraft industry is made up of a well organised formal sector that work with rural women in product development and exports most of its products. There is also an informal sector that mainly sells its products locally at tourist attraction sites. A wide range of products are made from natural fibres and serve as the rural women's source of livelihood. These products include handicrafts like baskets,

door mats, sleeping mats, wall hangings, drying mats or trays as packages for fruits and vegetables in local communities (Zwane and Masarirambi, 2009; Zwane *et al.*, 2011). Sale of fibre products contributes towards the livelihoods of communities. A little has been done to introduce fibres such as sisal in textiles where they also hold a potential for use. Sisal fibres are not widely used in textile development due to their coarseness and strength, however, they can be blended with cotton to produce yarns and fabric with a lesser harsh handle. To be effectively blended with other materials the fibres require some degree of chemical modification. Further, the aesthetic value of handicrafts relies greatly on colour, making dyeing a very important part of the trade. This study aimed at exploring the use of enzymes as an alternative method of sisal fibre surface modification for improved properties such as dyeability and pliability.

Literature summary

Fibre extraction can be achieved through mechanical and chemical processing. Mechanical means are usually utilized by rural communities, where rural women use rudimentary tools like can lids or aluminium containers to manually extract fibres from the leaves. Alternatively, manual decorticating can be done through the use of a machine decorticator modified to have a rotor, which prevents the blockage of machine by pulp or fibre waste and does a satisfactory job (Boguslavsky *et al.*, 2007). Mechanical extraction methods are not efficient in removal of cementing compounds (calcium pectates) between fibres and allied polymers, mostly waxes, cellulose, hemicellulose, lignin and hydrocarbons. Chemical extraction methods are the next best alternative to ease the extraction of fibres, and can be achieved by using acids, alkaline and enzymes.

Enzymatic processing has not gained popularity particularly in industries of developing countries, despite being eco-friendly (Anandjiwala, 2006). Enzymes are very effective biological catalysts due to their specificity, resulting in much higher reaction rates as compared to chemically catalyzed reactions under ambient conditions. Enzymatic processing degrades the lignocellulosic complex in fibre swelling, lower the degree of polymerization and make fibres more pliable and softer (Zwane, 1997; Dutta *et al.*, 2000). A group of enzymes that include cellulose, pectinase and hemicelulase have been used to control hydrolysis of the constituents in jute and have made the fibres soft, more pliable and spinnable than untreated fibres (Dutta *et al.*, 2000; Michalak and Krucinka, 2004). Soft fibres are relatively easy to work with and products made from them are comfortable to wear.

Study description

Qualitative. The participatory rural appraisal (PRA) technique and a questionnaire were used to assess current practices and experienced challenges of fibre processors in two administrative regions of Swaziland. Two developed data collection instruments were validated and used to collect data through one-on-one interviews.

Quantitative. An experimental research design was the focus in the laboratory tests conducted. Fibre extraction technique found to be used by the fibre processors was used to

extract fibres from species of the agave plants, with the assistance from experienced fibre processors. As a pre-treatment step, the fibres were thoroughly washed with distilled water and dried in a vacuum oven pre-set at 80°C for 24 hours, after which raw fibres were sampled. They were then treated with diethyl ether: Hexane: Ethanol (2:2:1) followed by ethanol, a modification of Moran *et al.* (2008) method to ensure the removal of all polar and non-polar compounds which includes waxes and other extractives, such that only macro molecules or polymers remained.

After the pre-treatment, manually decorticated fibres were cut into almost powder-like consistency using clean scissors to increase surface area for enzyme treatment (Handa *et al.*, 2008). Fibres were treated with sodium hydroxide and enzymes. Miller (1959)'s Dinitrosalicylic (DNS) method was used for monitoring hydrolysis and optimizing the enzymes. Two hemicellulase enzymes supplied by Novozymes, were used on sisal and the century plant with an aim of choosing a better performing one (high rate constant, *k*).

With the pineapple and another set of sisal fibres, two chemical pretreatments using ionic liquid (IL) and organic solvents were used for DNS assay and subsequent analysis respectively. For DNS assay 1-allyl-3-methylimidazolium chloride, [AMIM]Cl purchased from Rochelle Chemicals in South Africa, was used as a pretreatment. Even though ILs are considered a better alternative to organic solvents, the amount available was not enough to finish the work, therefore organosolv method using soxhlet apparatus was used in subsequent pretreatments (Handa *et al.*, 2008).

First optimization of enzyme treatment was carried out using 3,5-Dinitrosalicylic acid (DNS) assay method by (Jeffries and Ratledge, 1994). Alkali and enzyme hydrolysis procedures were then conducted to remove binding components in the lignocellulosic matrix. Sample fibres were soaked in beakers containing 5% NaOH and placed in a water bath controlled at 20±2 °C for 24 hours as adopted from Payae and Lopattananon (2009). Other sisal fibres were treated with pectinase, and lignin peroxidase enzymes. Conventional alkali treatment using 5% NaOH was used as a reference.

Enzyme treatments were conducted in a stepwise manner starting with lignin, followed by hemicellulase and then pectinase using similar conditions. The enzyme to buffer volume ratio was 2:8 v/v making 10 mL mixtures used to treat 1 gram fibres. A control was also included in this treatment where 1 gram fibres in 10mL buffer solutions only, was exposed to same temperatures and treatment time. After fibre treatment, chemical and mechanical properties of raw and treated fibres were analyzed using the FTIR spectroscopy, Scanning Electron Microscopy (SEM) and Instron Tensile Tester.

Indigenous dye substrates were harvested from the bark of *Sclerocarya birrea (umganu)*, *Syzigium cordatum (umcozi)* and *Berchemia zeyheri (umneyi)* trees. Aqueous extracts were then prepared by adding a 250g of ground bark in 1L distilled water. The mixtures were kept at room temperature for a day and then boiled for 3 hours. Equal samples of alkali treated fibres, enzyme treated fibres and untreated fibres were dyed with unfiltered liquors. Treated and untreated fibres were also dyed with one gram Astrazon yellow 8GL commercial

dye dissolved in one litre distilled water to make 1 mg/mL solution. For 10mL dye solutions 0.2 grams fibres were added and boiled for 1 hour at 60°C. Dyeability samples were prepared in triplicates for each variable test conducted. The calculation of dye exhaustion or absorbance of the dye bath before and after dyeing at the wavelength of maximum absorbance (λ_{\max}) of the dye used followed the method stipulated in Sricharussin *et al.* (2009). To improve the tactile properties of sisal products, an epoxy and polyester type of resin was applied following the resin casting method for encasing sisal fibre mats.

Obtained dye solutions were analyzed using a Shimadzu UV-VIS Spectrophotometer for absorbance. A completely randomized design with two factors was conducted on absorbance results using an ANOVA (F-test) at $\alpha=0.05$.

Research application

Findings of the qualitative component of the study found that all interviewed fibre processors were relying on raw materials in the wild and they were using mechanical and crude methods for decorticating the fibres (Mkhonta *et al.*, 2014; Vilane *et al.*, 2014). Fibre processors were only using sisal fibres and the production was mostly done by females (95%), some of whom were family providers. A monthly income of less than \$40 was made from selling sisal products which included handbags. It was however, found that the coarse nature caused difficulty in making jewellery (ear rings for example), hence the need of laboratory modifications with enzymes.

For sisal, enzyme kinetics demonstrated that hydrolysis occurred in a first order reaction with rate constant $k = 1.9 \times 10^{-3} \text{hr}^{-1}$ with V_{\max} decreasing in time. Infrared Spectroscopy results, showed that peak intensities at 2900cm^{-1} and 1735cm^{-1} and this corresponded with C—H and C=O vibrations of carboxylic acids or ester groups, respectively. The corresponding hemicellulose and pectin were reduced especially in NaOH.

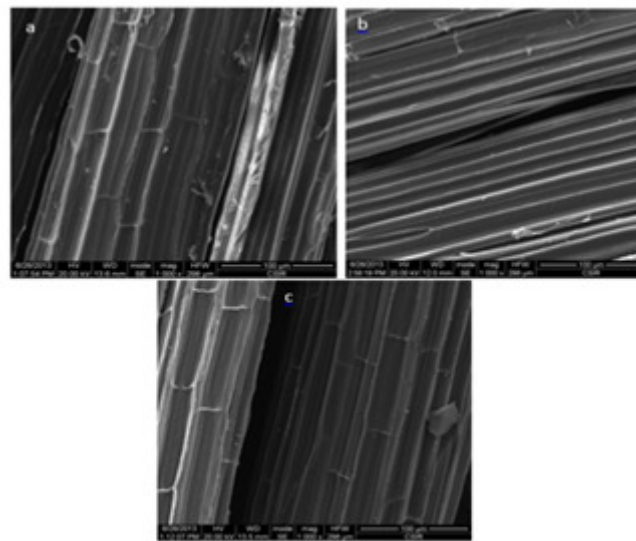
Table 1 shows that the alkaline treatment reduced sisal tensile strength by 32% while enzyme reduced it by 19%. Similar results were observed in *A. americana*, 34% with NaOH and 13% with enzyme. Also, NaOH reduced fibre diameter by approximately 25% as opposed to the enzyme at 16% in sisal (Table 2). Scanning electron microscope micrograms showed smoother enzyme-treated fibres than NaOH-treated ones (Fig. 1). Enzyme treated fibres improved dyeability compared to raw fibres. Hemicellulase and sequential treatment with all three enzymes (Ems) showed the best results in dyeability ($p < 0.05$). More dye was absorbed by fibres treated with alkali producing darker shades than those treated with enzymes. Lighter shades were observed for untreated fibres. The FTIR revealed that more hydroxyl sites opened as a result of treatment, hence the decrease in smooth trough transmittance at 3421cm^{-1} region (Fig. 2). The peaks at 3421 and 1035cm^{-1} are indicative of O-H stretching and O-H bending groups respectively. Free hydroxyl groups are a prerequisite to dye binding in lignocellulose fibres. Sequential treatments with all enzymes (Ems) were almost as effective as alkali treatment in opening free hydroxyl groups.

Table 1. Tensile strength of sisal before and after treatment

Sample name	Extension at max. load (mm)	Tensile stress at break (MPa)
AS-Raw	0.7	237
AS-NaOH	0.8	162
AS-Conc.L	1.3	192

Table 2. Fibre diameter before and after treatment

Sample #	Fibre diameter (μ)	CV (%)
AA-Raw	102.7	35.3
AA-NaOH	74.1	57.5
AA-Enzyme	91.2	32.1
AS-Raw	117.9	44.1
AS-NaOH	89.8	51.2
AS-Enzyme	97.4	32.6

**Figure 1. SEM micrograms of *A. sisalana* a) Raw b) Enzyme, and c) NaOH treated fibres**

Pineapple raw fibres had lower tensile strength than the sisal raw fibres which agrees with the findings of Reddy and Young (2005) that pineapple has lower mechanical strength hence higher pliability. Casting did not yield best result; it was too stiff, brittle and unusable. Further work is needed to improve the texture of the sisal pendants on jewellery. A light coating can be tried like a varnish instead.

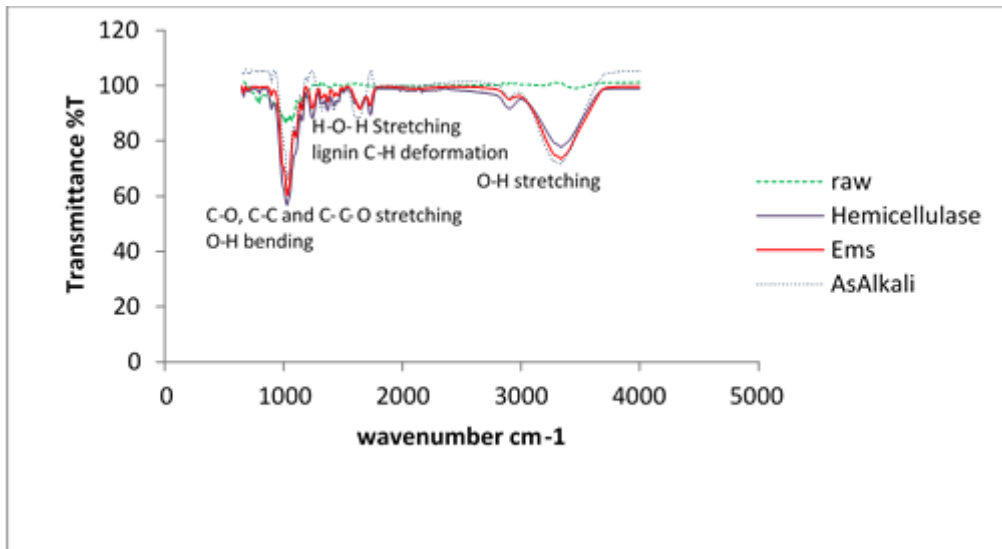


Figure 2. FTIR spectra of sisal fibres



Figure 3. Non-woven structure made from sisal fibres

Nonwoven development. Three samples of nonwoven material were made from raw fibre, control fibre and enzyme treated fibre at the Council for Scientific and Industrial Research (CSIR) in Port Elizabeth, South Africa (Fig. 3). Products like mirror supports and tissue box covers were explored using the nonwoven structure.

Conclusion

In this study, it was found that fibre processors were only using sisal fibres yet other complimentary fibres can be used when the sisal fibre is in short supply. Also the lack of appropriate technology to increase productivity of fibres was negatively affecting the work

of the women entrepreneur. This could be addressed through the use of an industrial decorticator. There was no significant difference between *A. sisalana* and *A. americana* fibre properties. On the other hand, pineapple fibres do not need enzymatic treatment but are naturally soft for use in product development. Enzymes were effective in reducing strengthened, fibre diameter and did not drastically compromise on the strength of fibre in product development. Sequential treatments with all enzymes (Ems) were almost as effective as alkali treatment in loosening the cementing layers between fibres and opening free hydroxyl groups, thus allowing more dye absorbance into the fibre structure. The advantage with the procedure is that it is not an environmental threat like the alkaline treatment currently being used. Recovery mechanisms of enzymes for reuse could be explored and this could offset their initial high purchase cost. Non-woven structures were successfully made from sisal fibres and new product development ideas were explored. There is need for more work on resin application to improve the hand of sisal pendants used in making jewellery.

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