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Development of an Integrated Dilute Acid-Lime Pretreatment of Chili Post-Harvest Residue for Bioethanol Production

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ABSTRACT : Lignocellulosic biomass serves as a renewable substrate for the production of biofuels and other value added products. It is composed of cellulose, hemicelluloses and lignin which are connected by several intra- and interpolymer linkages. Hence some kind of pretreatment to be carried out to make it accessible for enzymatic saccharification. Conventional pretreatment of lignocellulosic biomass is carried out by using either acid or alkali. During acid pretreatment, hemicelluloses is removed from the biomass in the liquid stream while in alkali pretreatment lignin is removed from the biomass in the liquid stream. An integrated process employing the benefits of acid and alkali pretreatment followed by enzymatic hydrolysis would be promising for better hemicelluloses and lignin removal. Chili post-harvest residue is an underexploited lignocellulosic biomass and India has an availability of 0.5 million tons of this residue. Till date only few reports were available for its utilization for the production of bioethanol and other value added products. Some kind of pretreatment to be carried to make it accessible for enzymatic saccharification by removing hemicelluloses and lignin. Though several conventional as well as alternative strategies have been developed addressing biomass recalcitrance, most of them are a failure or economically nonviable as an industrial process. The aim of the study was to develop an integrated dilute acid-lime pretreatment strategy for better hemicelluloses and lignin removal from the chili post-harvest residue for the production of bioethanol and to optimize various process parameters affecting pretreatment as well as characterization of the native and pretreated samples.

KEYWORDS : bioethanol, pretreatment, saccharification, acid, chili

NOMENCLATURE

CPHR	Chili post-harvest residue
IDALP	Integrated dilute acid lime pretreatment

I. INTRODUCTION

Depletion of fossil fuels and increase in environmental concerns like greenhouse gas emissions leads to search for alternative strategies of energy. Bioethanol serves as a potential alternative fuel. Lignocellulosic biomass serves as a potential renewable substrate for the production of bioethanol (Rezania et al., 2017). Usage of fossil fuels in automobiles release large amount of CO₂ which is a major cause of global warming.

Production of bioethanol from lignocellulosic biomass involves several unit operations like pretreatment, neutralization, enzymatic saccharification, detoxification and fermentation. One of the most energy intensive process is the pretreatment. Several research and developmental activities are going on in this direction to develop strategies which are economically viable. Most of the strategies currently in practice are energy intensive and economically nonviable.

The benefits of combined acid and alkali pretreatment was earlier reported by Zhou et al., 2013 for pretreatment of spent mushroom substrate for reducing sugar and biofertilizer production. The objective of the present study was to select the best dilute acid for integrated dilute acid lime pretreatment of chili post-harvest residue (CPHR) and to optimize various process parameters affecting integrated dilute acid –lime pretreatment of CPHR (IDALP CPHR) and utilization of the hydrolyzate obtained after enzymatic saccharification for the production of bioethanol as well as characterization of native and the pretreated samples.

II. MATERIALS AND METHODS

Feed stock

Chili post-harvest residue (CPHR) received from Virudhanagar, Tamil Nadu, India was used in this study. The samples were dried and milled using a knife mill.

Screening of various acids for integrated dilute acid - lime pretreatment of chili post-harvest residue (IDALP CPHR)

Initial screening experiments were carried out with four different acids (H_2SO_4 , HCl , HNO_3 and H_3PO_4) at an initial concentration of 2% w/w, biomass (solid) loading of 10% w/w and $\text{Ca}(\text{OH})_2$ concentration of 2% w/w.

Pretreatment carried out in a laboratory autoclave at 121°C for 60 min. After pretreatment the samples were used for hydrolysis after washing and drying.

Optimization of various process parameters affecting integrated dilute acid - lime pretreatment of chili postharvest residue

Optimization of various process parameters affecting integrated dilute acid-lime pretreatment of CPHR was carried out by adopting a Taguchi design. The experiment consists of a total of 16 runs. The parameters selected were biomass (solid) loading, HNO_3 concentration, $\text{Ca}(\text{OH})_2$ concentration and pretreatment time. Parameters like biomass loading, HNO_3 concentration, $\text{Ca}(\text{OH})_2$ concentration and pretreatment time were selected at four levels.

Compositional analysis, characterization of native and pretreated biomass by SEM, XRD and FTIR analysis

Compositional analysis of native and pretreated samples was carried out by adopting NREL protocol (Sluiter et al., 2008). To investigate physical and chemical changes of lignocellulosic biomass before and after pretreatment, characterizations were performed. In addition to compositional data, physical attributions like morphological changes, cellulose crystallinity changes and FTIR responses were also monitored.

III. RESULTS AND DISCUSSION

Compositional analysis of native and pretreated chili post-harvest residue

Compositional analysis of the biomass revealed that the native biomass contains 39.95% cellulose, 17.85% hemicelluloses and 25.32% lignin. Control 1 (water alone) contains 41.05% of cellulose, 16.79% of hemicelluloses and 24.11% of lignin. IDALP CPHR contains 43.89% of cellulose, 10.27% of hemicelluloses and 14.37% of lignin. Mass balance analysis revealed a 34% loss of biomass during the pretreatment process. IDALP was found to be effective in removing hemicelluloses and lignin.

Screening profile of various acids for integrated dilute acid - lime pretreatment of chili post-harvest residue

Four different acids – H_2SO_4 , HCl , HNO_3 and H_3PO_4 at 2% w/w were used for initial screening to select the best acid for IDALP of CPHR. Control experiments were carried out with water alone Initial screening were carried out with 10% w/w of biomass (solid) loading, dilute acid concentration of 2% w/w, lime concentration of 2%w/w and pretreatment time of 60 min in a laboratory autoclave at 121°C . Control samples were the pretreatment carried with water alone gave a reducing sugar yield of 0.05 g/g. H_2SO_4 , HCl , HNO_3 and H_3PO_4 assisted lime pretreatment gave a reducing sugar yield of 0.253, 0.214, 0.284 and 0.204 g/g respectively. IDALP CPHR gave a better reducing sugar yield when compared to dilute acid pretreated alone or lime pretreated alone samples. Since HNO_3 assisted $\text{Ca}(\text{OH})_2$ pretreated samples gave higher reducing sugar yield it was selected for further optimization of different process parameters affecting IDALP of CPHR by adopting a Taguchi design.

Effect of different process parameters on IDALP of chili post-harvest residue

Maximum reducing sugar yield (0.622 g/g) was observed in Run No: 6 where the conditions of pretreatment were $\text{Ca}(\text{OH})_2$ concentration of 2% w/w, HNO_3 concentration of 5% w/w, biomass loading of 10% w/w and pretreatment time for 30 min.

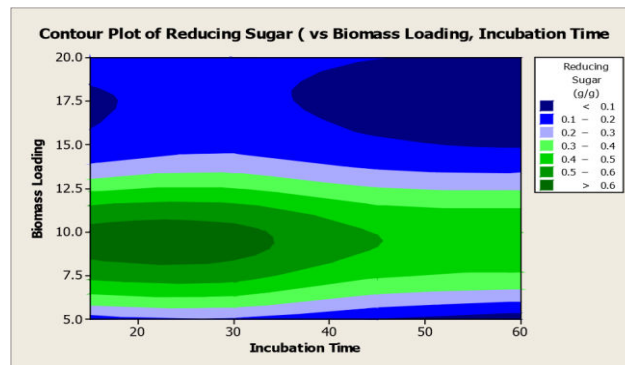


Fig.1. Contour plots showing interaction between pretreatment time and biomass loading on reducing sugar yield

Fig. 1 shows an interaction between pretreatment time and biomass loading. At low to middle levels of pretreatment time (15 – 30 min) the reducing sugar yield is high (0.60 g/g). It decreases with increase of pretreatment time (40–60 min). At low levels of biomass loading (5.0 – 7.5% w/w) the reducing sugar yield is low (0.40 g/g); it increases with increase of increase of biomass loading (7.5–11.5%w/w). Maximum reducing sugar yield (0.60 g/g) was observed with middle levels of biomass loading (9.5–10% w/w) and low to middle levels of pretreatment time (15–30 min).

Characterization of native and pretreated biomass by SEM, XRD and FTIR

Characterization of native and pretreated biomass were carried out by SEM, XRD and FTIR. The results indicate that there is difference between the native and pretreated samples. Fig.2 shows the scanning electron micrographs of native, hydrothermal, dilute HNO_3 , $\text{Ca}(\text{OH})_2$ and IDALP CPHR. The native samples showed a highly ordered and compact structure while the pretreated samples showed a highly distorted structure.

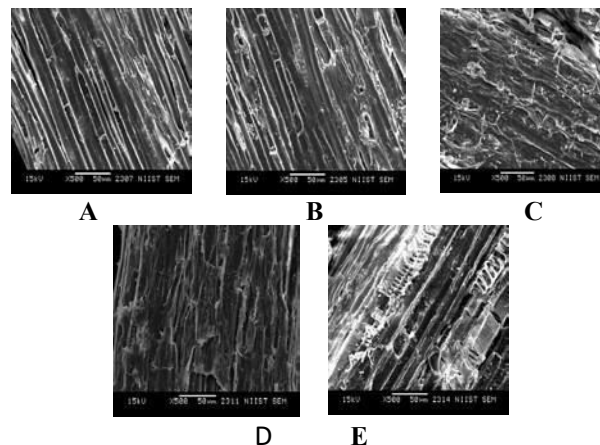


Fig.2. Scanning electron micrographs of native and pretreated samples (A-Native, B-Hydrothermal, C- Nitric acid, D- Lime and E-integrated dilute acid-lime)

IV. CONCLUSION

Compositional analysis data revealed that hemicelluloses and lignin were removed during IDALP of CPHR. Maximum reducing sugar yield of 0.622 g/g was observed after optimization of various process parameters affecting IDALP of CPHR. To the best of our knowledge, this is the first report on IDALP of CPHR. Fine tuning of various process parameters will make the process economically viable.

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