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Pre-Treatment Process for Sorghum Biomass for Preparation of Bio-Ethanol

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I. INTRODUCTION

A. *BIO-MASS*

Biomass is the term used for the biological material from living or recently living organisms such as wood, waste materials, gases and alcohol fuels. It is commonly plant matter that is specifically grown in order to produce electricity or to produce heat. The composition of biomass is carbon, hydrogen oxygen, nitrogen and small quantities of other atoms, including alkali, alkaline earth and heavy metals can also be found. Biomass is the building block or 'feedstock' for many other fuels. It refers specifically to lingo-cellulose in abundance. Cellulose, hemicelluloses, and lignin are its major constituents.

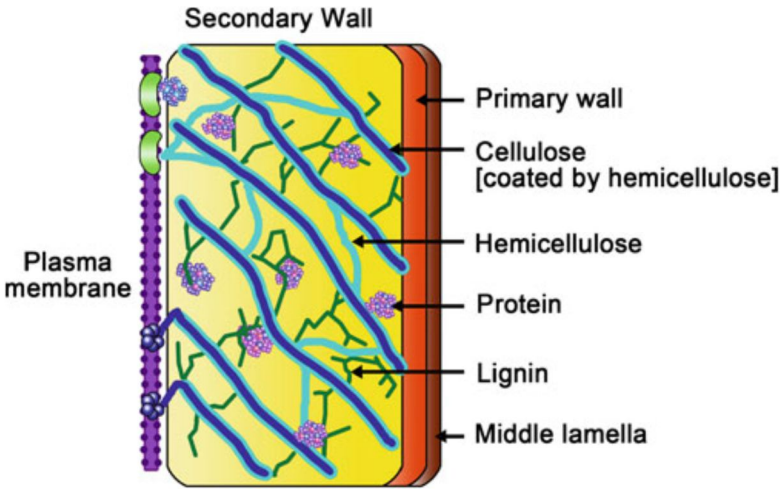
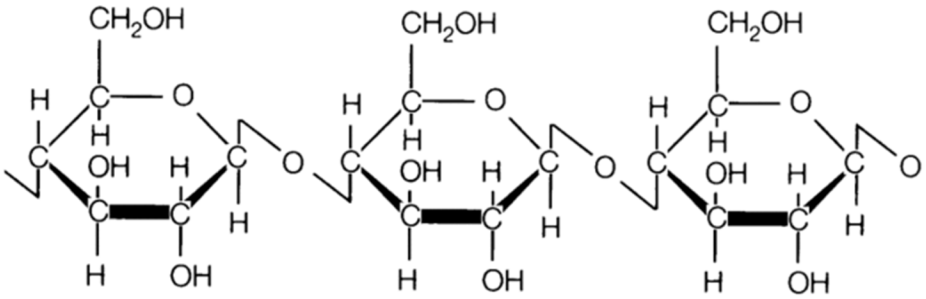


Fig- Internal structure of biomass

B. *Major Components Ofbiomass*

1) *Cellulose:* Cellulose is an organic compound with the formula $(C_6H_{10}O_5)_n$, a polysaccharide consisting of a linear chain consisting of D-glucose subunits linked to each other by β-(1, 4)-glycosidic bonds. Cellobiose is the repeated unit established through this linkage, and it constitutes cellulose chains. The long-chain cellulose polymers are linked together by hydrogen and vanderwaals bonds, which cause the cellulose to be packed into micro fibrils.



2) *Hemi-Cellulose:* It is a complex, branched and heterogeneous polymeric network, based on pentoses such as xylose and arbinose, hexoses such as glucose, mannose and galactose, and sugar acids. It has a lower molecular weight than cellulose and its role is to connect ligno and cellulose fibres. The amount if hemicellulose is usually between 11% and 37% of the lingo-

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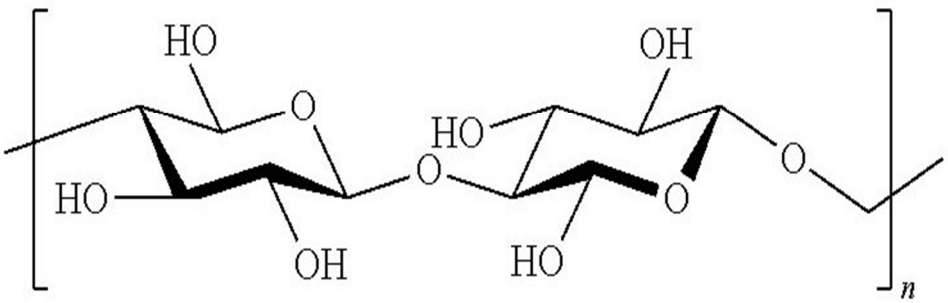
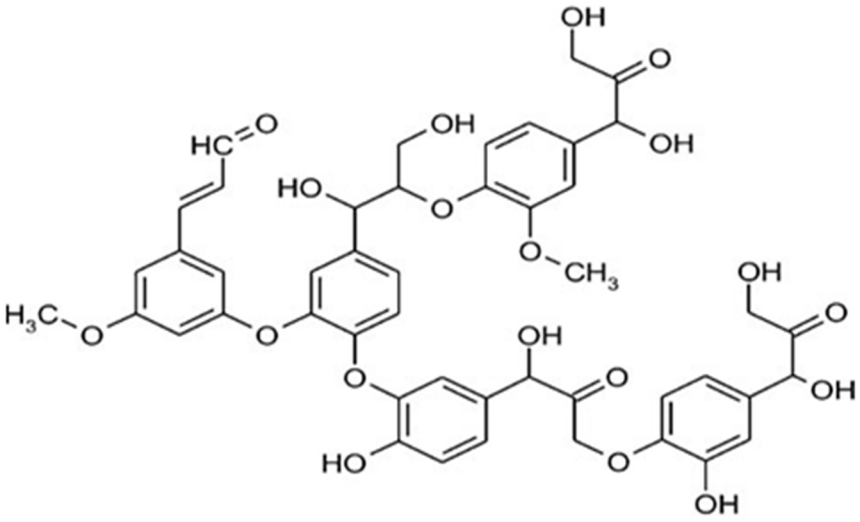
cellulosic dry weight. Hemicelluloses are embedded and interact with cellulose and lignin, which significantly increase the strength and toughness of plant cell walls.

a) *Structure of hemi-cellulose*:



Hemicellulose

b) *Lignin:* It is a very complex molecule constructed of phenyl propane units linked in a three- dimensional structure. Generally, softwoods contain more lignin than hardwoods. It (15–25% of total feedstock dry matter) is an aromatic polymer synthesized from phenyl propanoid precursors. Lignin is hydrophobic and highly resistant to chemical and biological degradation. It is present in the middle lamella and acts as cement between the plant cells. It is also located in the layers of the cell walls, forming, together with hemicelluloses, an amorphous matrix protecting against biodegradation.



Lignin

C. *Types of biomass:* Most abundantly used biomass as raw material till date is wood. For most industrial purposes corn, poplar, willow, sorghum, sugarcane, bamboo, and a variety of tree species ranging from eucalyptus to oil palm are cultivated and used. However crop residues like cane bagasse, corn stover, wheat straw, rice straw, rice hulls, barley straw, sweet sorghum bagasse, olive stones and Pulp, hardwood (aspen, poplar), softwood (pine, spruce); cellulose wastes (newsprint, waste paper, recycled paper) and municipal solid wastes (MSW) are also different kinds of biomass.

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Figure- various biomasses

In recent years the concept of the bio-refinery has emerged, whereby one integrates biomass conversion processes and technology to produce a variety of products including fuels, power, chemicals and feed for cattle. In this manner one can take advantage of the natural differences in the chemical and structural composition of the biomass feed stocks. There have been two different generations of biomass. For the conversion of biomass to fuel, the biomass needs to undergo the following three steps they are:.

1) *Pre-Treatment Using Various Techniques:* Pre-treatment methods can be roughly divided into different categories:
a) Physical (milling and grinding)
b) Physicochemical (steam pre-treatment/auto-hydrolysis, hydrothermolysis, and wet oxidation)
c) Chemical (alkali, dilute acid, oxidizing agents, and organic solvents), biological, electrical, or a combination of these.
2) *SACCHARIFICATION:* This is in general hydrolysis which refers to the process that converts the polysaccharides into monomeric sugars. Sample of pre-treated raw material is added with a buffer solution, enzyme and subjected to incubator at prescribed temperature conditions to break down cellulose and form simple sugars like glucose, fructose etc.
3) *Fermentation:* Ethanol fermentation is a biological process in which organic material is converted by microorganisms to simpler compounds, such as alcohols. These fermentable compounds are then fermented by microorganisms to produce ethanol and CO_2.

II. EQUIPMENTS

A. Size Reduction Unit

Reduction of particle size is an important operation in many chemical and other industries. The important reasons for size reduction

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are:

1) Easy handling
2) Increase in surface area per unit volume
3) Separation of entrapped components

The operation is highly energy intensive; hence a variety of specialized equipment is available for specific applications. The equipment may utilize one or more of the following physical mechanisms for size reduction:
(i) Compression, (ii) Impact, (iii) Attrition, (iv) Cutting. Estimation of energy for the operation is important and is usually done by empirical equations. Enormous quantities of energy are consumed in size reduction operations. Size reduction is the most inefficient unit operations in terms of energy, as 99% of the energy supplied goes to operating the equipment and producing undesirable heat and noise, while less than 1% goes in creating new interfacial area. Reduction to very fine sizes is much more costly in terms of energy as compared to relatively coarse products.

Figure: size reduction unit

B. Steam explosion unit

Raw material is heated up to a temperature range of 180□C with a pressure range of 10Kg/cm^2 followed by explosive decompression resulting rupture of rigid structure of biomass.

Figure- Steam explosion unit

C. Vacuum filteration unit

In the filtration process the reaction mixture after pre-treatment was subjected to vacuum filtration where the biomass is filtered and further subjected to water wash in order to neutralize the feed for saccharification.

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Figure- vacuum filtration unit

D. Soxhlet extraction unit

This process was mainly used in extraction process. In this process the sample was subjected for extraction using soxhlet extraction to calculate the amount of water and ethanol extractives present in raw and pre-treated materials.

Figure- Soxhlet extraction

1) Solvent table

Solvent	Formula	Molar mass in g/mol	Evaporation Energy in J/g	B.P at 1013 mbar	Density in g/cm^3	Vacuum in mbar for B.P at 40^0c
Methanol	CH_40	32.0	1227	65	0.791	337
Water	H_20	18.0	2261	100	1.000	72

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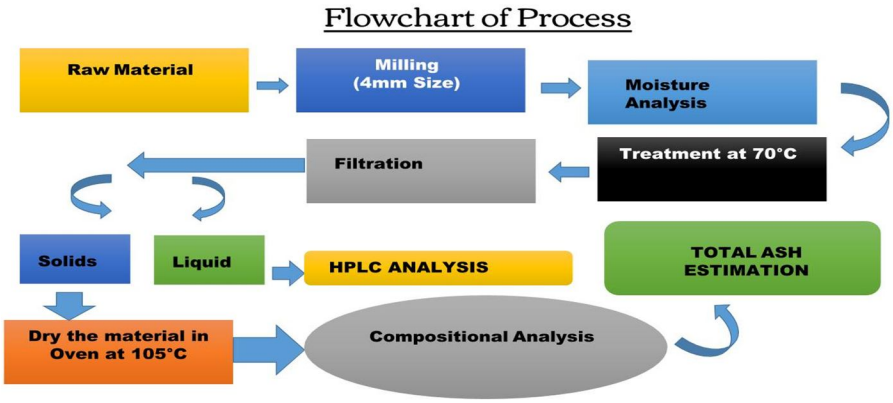
III. MATERIALS & METHODOLOGY

A. *Pre-treatment Of Bio-Ethanol*



1) *Raw material:* Here we used sweet sorghum as a raw material for the pre-treatment processes of biomass.
2) *SORGHUM:* Sorghum is a potential feedstock for second generation bio-ethanol. Sorghum is a genus of plants in the grass family. Most species are native to Australia, with some extending to Africa, Asia, Mesoamerica, and certain islands in the Indian and Pacific Oceans One species is grown for grain and many of which are used as fodder plants, either cultivated or as part of pasture. The plants are cultivated in warm climates worldwide and naturalized in many places. Sorghum is in the subfamily Panicoideae and the tribe Andropogoneae (the tribe of big bluestem and sugarcane). The leaves of sorghum and maize are similar but in the case of sorghum they are covered by a waxy coat that protects the plant from prolonged droughts. The sorghum grain is grouped in panicles and the plant height ranges from 120 to 400 cm depending on type of cultivar and growing conditions. An advantage of sorghum compared to maize is that it has a comparatively lower seed requirement because only 10 to 15 kg/ha are used compared with 40 kg/ha required by other cereals. The mature stems of sweet sorghum contain about 73% moisture and the solids are divided in structural and non-structural carbohydrates. Approximately 13% are non-structural carbohydrates composed of sucrose, glucose and fructose, in variable amounts according to cultivar, harvesting season, maturity stage, among other agronomic factors. Compared to sugar cane, the main difference is that the sucrose content in cane is significantly higher compared to glucose and fructose (90, 4 and 6%respectively) and the total content sugar is 49% of the dry stem weight. In general terms, composition of simple sugars in sweet sorghum juice is 53-85, 9-33 and 6-21% for sucrose, glucose and fructose, respectively. Sweet sorghum has 13% fiber, 44.6% cellulose, 27.1% hemi-cellulose, 20.7% lignin, 0.4% ash.

Fig- Sorghum plant

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3) Methodology: The goal of the pre-treatment process is to remove maximum lignin and hemi-cellulose content, reduce the crystallinity of cellulose, and increase the porosity of the ligno-cellulosic materials.

Pre-treatment methods can be roughly divided into different categories: physical (milling and grinding), chemical (alkali, dilute acid, oxidizing agents, and organic solvents), biological, electrical, or a combination of these. The following pre-treatment technologies have promise for cost-effective pre-treatment of ligno-cellulosic biomass for biological conversion to fuels and chemicals.

B. Physical pre-treatment

Combination of ligno-cellulosic materials through a combination of chipping, grinding, and/or milling can be applied to reduce cellulose crystallinity. The size of the materials is usually 10-30 mm after chipping and 0.2-4 mm after milling or grinding.

Fig- Miller set at 700 rpm with 4mm sieve

Fig- Sweet sorghum biormass-10mmsize

Fig- milled sorghum-4mm size

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C. Chemical treatment

1) Alkali Peroxide Treatment

a) Solid mass was added with 3% NaOH and fed to jacketed reactor with drop-wise addition of 3% H_2O_2 after attaining the temperature to 70□C the reaction is carried out for 3 hrs.

b) The reaction mixture was filtered and washed with water to neutralise the solids. The solid and liquid samples were further subjected to component analysis and drying.

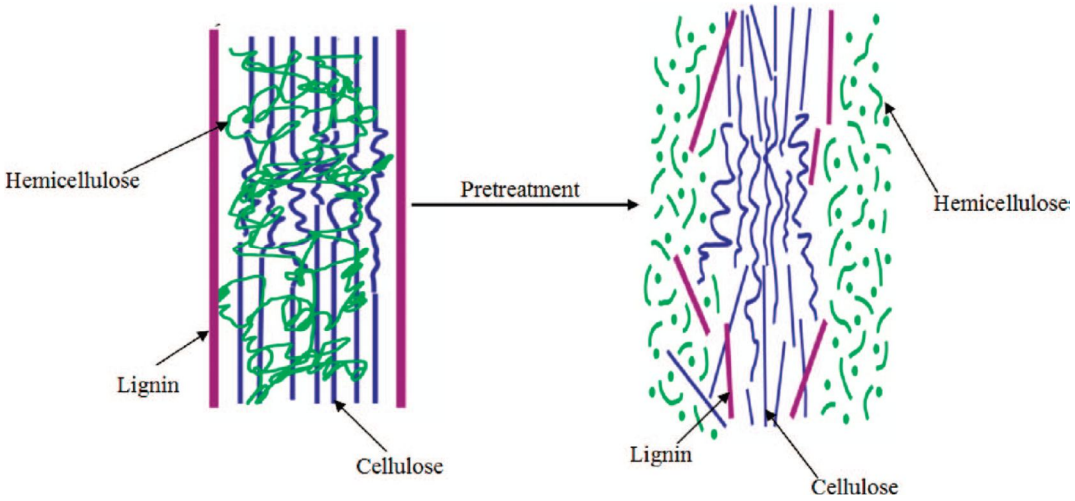

Fig- Before and after pre-treatment structure of biomass

IV. ANALYSIS

A. Estimation Of Lignin After Pre-Treatement

1) Procedure For Lignin Analysis

a) 300mg of dried bio-mass is taken in 500mL of screw capped bottle.

b) Add 3mL of 72% of H_2SO_4 (w/w) transfer into bottle.

c) All the samples are to be mixed rigorously for 1hr to ensure the acid to particle contact and uniform hydrolysis.

d) Upon completion of 1hr hydrolysis dilute to 4% (w/w) acid concentration by adding 84mL of distilled water and mix the quantities thoroughly.

e) Put the samples in autoclave for an hour at 121°C.

f) After autoclaving do not remove the caps immediately leave them to cool to room temperature.

g) The autoclave hydrolysis liquor by passing through the filtering crucibles.

h) The solid will retain on the crucible and at the bottom filtrate will obtain.

i) The filtrate received from vacuum filter is sent for analysis in UV spectrophotometer.

Here the percentage of acid soluble lignin is calculated by:

$$\%ASL = \frac{UV_{abs} \text{ x volume of filtrate x dilution}}{€ \text{ x } OD_{Wsample} \text{ x path length}}$$

Were UV_{abs} = Average absorbance for the sample at appropriate wavelength.

$OD_{Wsample}$ = Oven dry weight

j) Solid mass is kept in muffle furnace at 575°C for 3hrs.

The weight of crucible before and after the muffle furnace is noted.

The percentage of Acid insoluble lignin is calculated by:

$$\% AIL = W_{cAIR} - W_{cASH}$$

W_{cAIR}=weight of crucible + acid soluble residue (AIR)

W_{cASH}=weight of crucible (after the heating) + ash

OD_{sample}=optical density of the sample.

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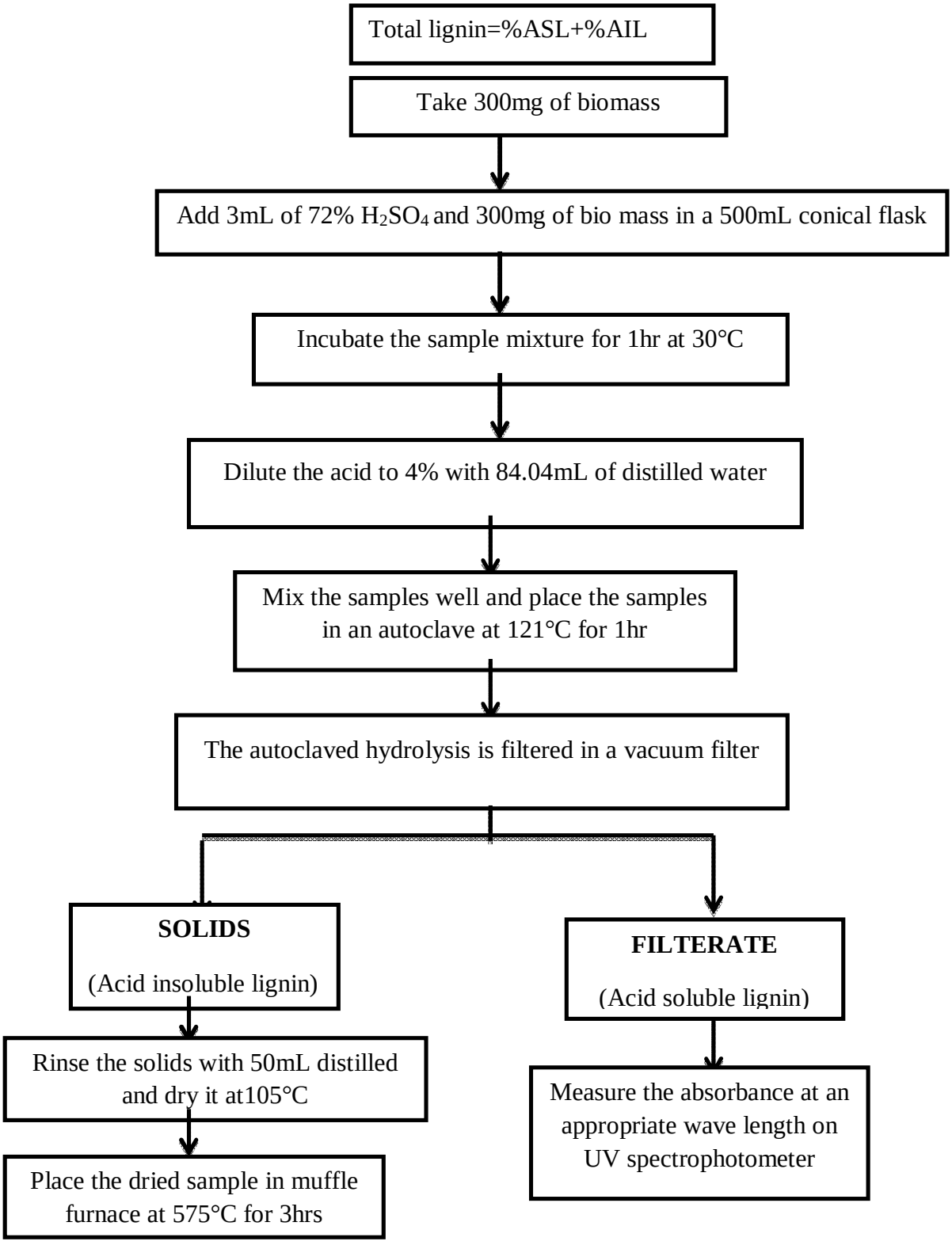
B. Schematic representation for determination of lignin inbio-mass



C. Calculations

1) Acid Soluble Lignin

%ASL = UV_{abs} x volume of filtrate x dilution

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$$\frac{}{€ \text{ x OD } _{Wsample} \text{x path length}}$$

UV_{abs}=0.269
Volume of filtrate = 86.73mL
Dilution = 15 , € =25 ,$OD_{Wsample}$=300mg
Path length = 1cm , %Extractives = water +ethanol extractives = 6.35

$$\%ASL = \frac{0.269 \text{x } 86.73 \text{x } 15 \text{ x } 100}{25 \text{x } 300 \text{x } 1}$$

= 4.66 (extractive free basis)

$$\%ASL \text{ (Received)} = \%ASL \text{ x } \frac{(100-\%Extractives)}{100}$$

= 4.66 x (100-6.35)/100
= *4.364 (1)*

2) Acid insoluble lignin

% AIL = $W_{cAIR} - W_{cASH}$
W_{cAIR} =55.0287g
W_{cASH} = 54.9343g
% AIL = (55.0287 - 54.9343) x 100 = 9.44 (Extractive free)

$$\%AIL \text{ (Received)} = \%AIL \text{ x } \frac{(100-\%Extractives)}{100}$$

= 9.44 x (100-6.35)/100 =8.84 (2)
Total lignin = %ASL+%AIL = (1)+(2) = 4.364+8.84
TOTAL LIGNIN = 13.204g

D. Composition data of sorghum bio-mass before and after pre-treatment

EXPERIMENT-6:

S.NO	COMPONENTS	SORGHUM COMPOSITION	
		RAW MATERIAL	AFTER PRE-TREATMENT
1.	Cellulose	36.96	64.66
2.	Hemi-cellulose	23.21	18.17
3.	Lignin	22.66	5.64
4.	Ash	2.43	0.25
5.	extractives	14.58	6.27
		99.84	94.99

Alkali peroxide treatment for sweet sorghum biomass at 70□C in glass jacketed reactor

1) Input

FEED – 600 gm + 50 gm extra (as 10% moisture)
WATER – 6000 gm
NaOH – 15% on bio-mass basis (90 gm)

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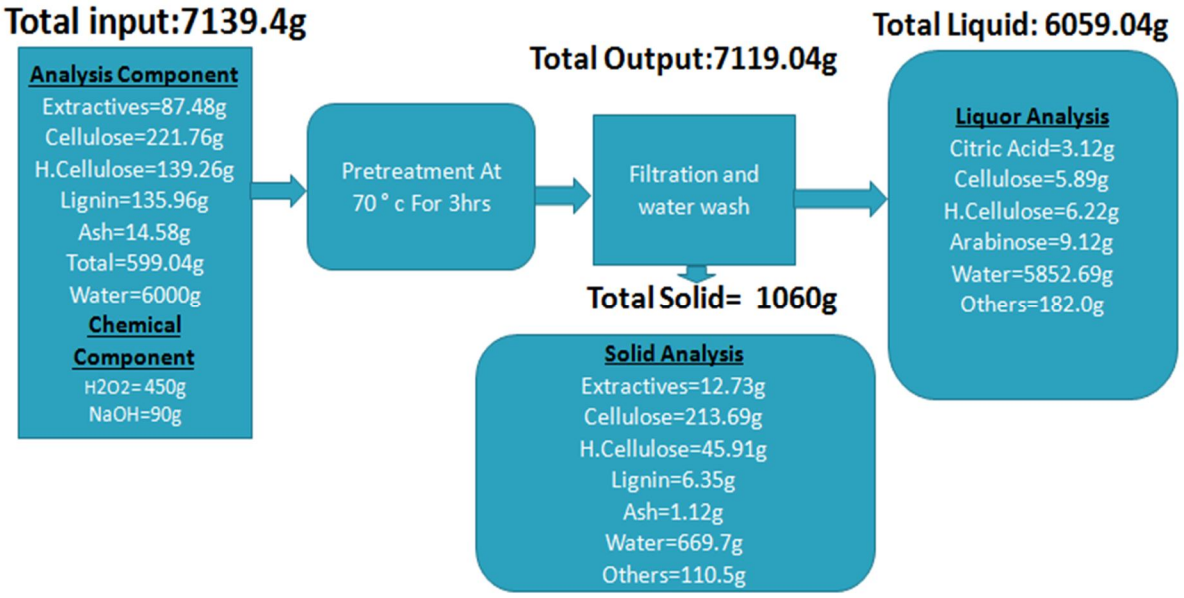
H_2O_2 - 20% on bio-mass basis (450 gm)
TOTAL – 7140 gm

2) Output
Weight of liquor = 6059.04 gm
Weight of biomass = 1060 gm (78% moisture, 22% solids)
TOTAL – 7119.04 gm

3) Loss
In – Out = 20.36gm



V. CONCLUSION

A. After conducting series of experiments for pre-treatment process maximum conversion of cellulose yield was obtained.
B. After conducting series of experiments, for pre-treatment of bio-mass which is a part of preparation of bio-ethanol concludes substantial conversion of cellulose was obtained up to 62%.

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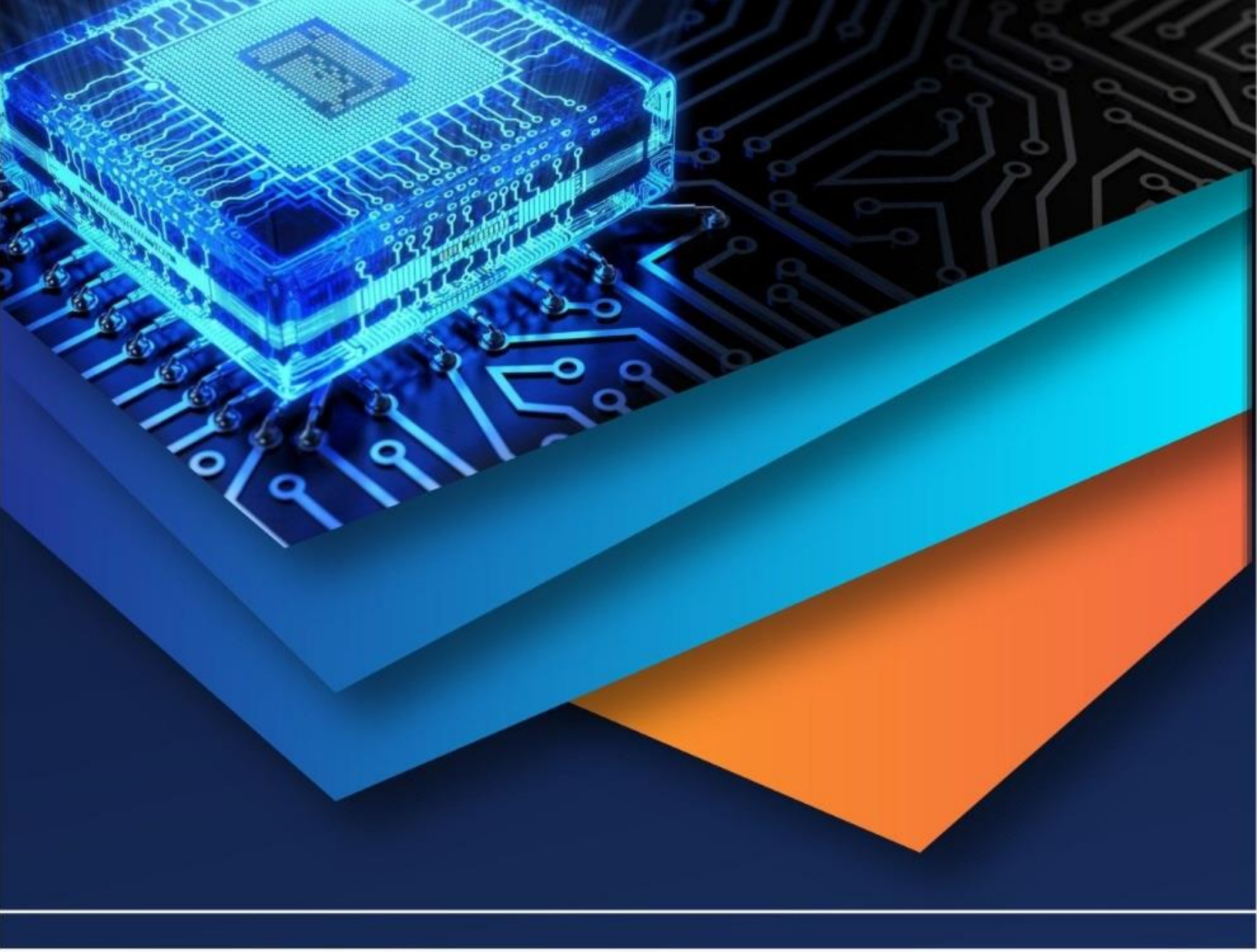

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