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StudyofCrystallinityIndexandThermalPropertiesofSweet
SorghumFiberafterPressurized-CookerTreatment
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Themainpurposeoftheresearchistoinvestigatethee?ectofthermal
treatmentthroughcommercialpressurecooker(5,25,and60min)onthermal
andcrystallinitypropertiesofstalksweetsorghum?bres.Crystallinityindex
andthermalstabilityofthe?bershavebeenstudiedbyusingX-raydi?raction
(XRD)andsimultaneousthermalanalysis(STA)characterization,respectively,
comparedtountreated?bers.Theresultofthisstudyshowsthatthis
treatmentincreasedcrystallinityindexandthermalstabilitypropertiesofthe
?bers.Thisresultalsoshowsthatsorghum?bercanbeusedfora
reinforcementcandidateinthermoplasticmatrix.

1.Introduction The strict regulations on the environment, and the limited re-
sourcesthatcomefromnature,encourageresearcherstolookfor
environmentallyfriendlyandrenewablematerials.Natural?ber
emergedasanalternativematerialthatissustainableandwidely availableinnature. At
present, natural ?bers are widely applied as ?llers and re- inforcement materials in
polymer composites for various appli- cations, at least in part, to replace glass
?bers.[1-6] This is due to some interesting properties, such as renewability, sustain-
ability,noimpactonglobalwarming,lowcost,biodegradability, nonabrasive properties
during processing, and well mechanical propertiescomparabletoglass?bers.[2]
Ontheotherhand,nat- ural?bershavemajorweaknessesthatarenaturallyhydrophilic so that
the bonding interface between natural ?bers with a hy- drophobic matrix is weak, low
maximum working temperature processing,andseasonal.[7] Several experiments have

been carried out to correct these de?ciencies, including modifying natural ?bers through chemical methods.

Based on reports from literature,[8-11]
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ChemicalEngineering UniversitasBhayangkaraJakartaRaya2ndCampus
Bekasi,WestJava17121,Indonesia DOI:10.1002/masy.201900129 alkalinization, acetylation, and bleaching treatments can reduce their hydrophilic properties, as a result the compatibility be- tweennatural?bersandhydrophobicpoly- mers increased. On the other hand, these methods are not environmentally friendly.

In this context, the motivation of this re- searchistostudythee?ectofthermaltreat- mentthroughcommercialpressurecooker, asthesimplestandgreenestprocess,[12] on changes in crystallinity index and thermal propertiesofstalksweetsorghum?bers. 2.ExperimentalSection 2.1.Materials The ?ber used in this research was the stalk sweet sorghum ?ber grounds.

They were obtained from Southeast Asian Min- isters of Education Organization—Tropical Biology (SEAMEO- BIOTROP)BogorinWestJavaIndonesia. 2.2.PreparationofSorghumFibers Sweetsorghum?bergroundwascutusingacrusherand?ltered usinga100mesh?lter.The?bersthatpassedfromthe?lterwere soaked in a pressure cooker (Oxone Ox-1060F with dimension 50 ×30 ×29cm),variationinsoakingtime(5,25,and60min).

After that, the ?ber sample was ?ltered and dried in a vacuum furnaceatatemperatureof50°Cfor120min. 2.3.Characterization 2.3.1.X-rayDi?raction(XRD) XRD was carried out using the Shimadzu XRD-7000 X-ray di?ractometer with a voltage of 40 kV and current of 20 mA using Cu-K ?? radiation, and the intensities were measured in the range of 5° < 2 ??ÿ< 40°.

The resulting di?ractogram was used to calculate the crystallinity index of untreated and treated sorghum?bers,Equation(1)wasinitiallyproposedbySegaletal in1959,[13] whichis: Cr I = (I002 - I am) / I002 ×100 % (1) where Cr Iisthecrystallinityindex, I002isthemaximumintensity ofthe(002)latticedi?raction,and I am istheintensitydi?raction oftheamorphousband.

 Figure1.

XRDdi?ractogramsfortheuntreatedandtreatedsorghum(SV: sorghum virgin/untreated sorghum; BP: pressurized-cooker; 5, 25, and 60minute).
2.3.2.ThermogravimetricAnalysis(TGA)
Thethermalstabilityofuntreatedandtreatedsorghumwasper- formed using simultaneous thermal analysis (STA) 6000/8000,
PerkinElmerinstrument.Allsampleswereheatedfrom40°Cto
500°Cataheatingrateof10°Cmin -1.Then,theexperimentwas
continuedfrom500°Cto800°C,atheatingrateof60°Cmin -1,to
burntheremainsoforganicmaterialduringtheheatingprocess.

2.4.ResultsandDiscussion X-ray di?ractograms for untreated and treated sorghum ?bers areshowninFigure1.The?gureshowesalmostthesameshape with three peaks of the crystalline plane. The peaks 1, 2, and 3,
locatedat2thetas16.1°,22.1°,and34°,areaccordingtocrystal-
lographyplaneof(10-1),(002),and(004),respectively.[14,15]Inthe literature,[16]
thepeakcontributiontothe?eld(004)isalmostir- relevant with the other two peaks.

Therefore, based on his re- search,the?rstandsecondintensitypeakscanbeanalyzedand consideredasanindicationofcellulosecontent. Cellulose consists of crystalline and amorphous structures. The strength of cellulose ?bers increases with increasing crys- talline content in cellulose, but on the other side their ?exibil- ity decreases.

The crystallinity index is an important parameter
indeterminingthecrystallinestructureportionincellulose.Ta- ble 1 shows the value of the untreated and treated crystallinity index of sorghum ?bers which were calculated using the Segal equation. Table 1 shows an increase in the value of the crys- tallinity index of ?bers from the pressurized-cooker treatment, compared to those untreated ?bers.

Increasing the crystallinity index value of the ?ber indicates that the structure of cellulose is more ordered or crystalline increases and the amorphous re- giondecreases. Whilethee?ectofthelengthoftreatmenttime on changes in the crystallinity index was not signi?cant, even Table 1. Comparison of crystallinity index between untreated and treated sorghum?bers. SV BP5 BP25 BP60 Iam 2620 2811 3023 2678 I002 5107 6205 6351 5839 CrI 48.7 54.7 52.4

54.1 appeared to be almost similar, and the presto-boiled treatment at5minshowedthehighestvaluewhencomparedwiththetime of 25 and 60 min. The duration of the treatment causes the de- structionofhydrogenbondsincelluloseduetothehydrolysisof excesswatermolecules,andthiscausesthecrystallineportionin cellulosetodecrease. The increase in the crystallinity index of ?bers subjected to pressurized-cooker treatment shows that the crystalline portion ofthe?berincreases,andontheotherhand,theamorphousde- creases. Figure 2shows the FE-SEM morphology of untreated and treated sorghum ?bers.

Images show that the pressurized- cooker treatment dissolves some of the amorphous content in ?bers,suchaswax,lignin,andhemicellulose.Itismarkedbypar- titionpatternalongthe?berbundlesurfaces.Duringthistreat- mentprocess,waterwillevaporateandbeabsorbedbysorghum ?bersandcausethe?berstobecomeswollenand?brillated. Thethermalpropertiesofnatural?bersareoneoftheimpor- tant parameters to be studied, especially their use as reinforce- mentinpolymercompositematerials.[17] Theprocessofmaking polymer composites involves the mixing process, natural ?bers as a reinforcement, and polymers as a matrix, at high tempera- tures with a speci?c time. In thermoplastic polymer materials, the mixing process is usually carried out at a temperature of around 200 °C.[15] Therefore, a good thermal stability is needed topreventthedegradationprocessofthe?ber,andtheresultwill impactthemechanicalpropertiesofcompositematerials.[15,17] Figure 3shows the degradation behavior of untreated and treatedsorghum?bers.AsshowninFigure3a,thermaldegrada- tionoccursinthreemainstages,forbothuntreatedandtreated sorghum?bers.Thisphenomenonisfoundandexplainedinde- tail in refs.[18-20].

In the ?rst stage, this occurs under a temper- ature of 100 °C. Losss of weight is caused by the dispersion of water content in the ?ber. The water contained in the ?ber can beintheformoffreewaterorlinkedwater.[6] Freewateriswater that sticks to the surface of natural ?bers and will evaporate at lower temperatures (25–150 °C), and linked water is water that formsbondswithhydroxylgroupsonlignin,hemicellulose,and cellulose,andevaporatesathighertemperatures.Afterfreewater evaporates,degradationinrangeof150—500°Coccursinlinked wateratlignin,hemicellulose,andcellulose.Thenextstageisre- latedtothethermaldegradationofhemicelluloseandsomepor- tionsoflignin.Itoccursatarangeof150–350°C.Finally,athigh- temperature range 310–400 °C, it shows the process of thermal degradation for cellulose and lignin, and almost cellulose com- ponent decomposed.[18-20] Lignin, because it consists of phenyl groups, has a wide temperature degradation range of 250–600 °C [18] or200–700°C,[20] whencomparedwithhemicelluloseand cellulose.

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Figure2. FE-SEMsorghum?ber:a)SV,b)BP5,c)BP25,andd)BP60. Related to the results of this study, as shown in Figure 3b, it showsindetailthethermaldegradationofsorghum?bersinthe temperature range of 100–300 °C. The ?gure shows the stabil- ity of sorghum ?bers under pressurized-cooker treatment was better when compared to untreated ?bers.

The initial temper- ature degraded ?ber (T i), a sample loses 3% of its weight,[18] BP5sorghum?berhasthebestthermalstabilitywhencompared with BP25, BP60, and untreated ?bers. The highest values of T i for ?bers are BP 5, BP25, SV, and 60, of which the highest are 251, 64, 60, and 59 °C, respectively. The high value of T i in BP5 ?bers may be associated with low volatility and hemicellu- lose.Figure3ashowsthenextthermaldegradationatarangeof 240–390°C.Theseresultsclearlyshowthatthethermalstability ofsorghum?bersunderpressurized-cookertreatmentishigher than untreated sorghum ?bers.

Higher crystallinity index value indicatestheportionandtheregularityofcellulosestructurein- creasesandbecomesdenser,whichmakestheheattransferdif- ?cult. The di?culty of heat transfer in the ?ber causes higher thermalstability.[17,20] 3.Conclusion The e?ect of thermal treatment of sorghum ?ber through pressurized-cooker treatment, variations of heating time, changes in ?ber structure was carried out in this study.

The observations of XRD showed that sorghum ?bers, which were modi?ed by pressurized-cooker treatment, contained more regular cellulose content (crystalline) compared to untreated sorghum?bers.Thisisprovenwithincreasinginthecrystallinity index of the sorghum ?ber after treatment. The increased in the crystallinity index of the ?ber, slows down the degrada- tion process, and increases the thermal stability of sorghum ?bers.

Acknowledgements The authors thank for the support from DRPM UI through its program PIT9GrantSchemewhichmadethisresearchpossible. Con?ictofInterest Theauthorsdeclarenocon?ictofinterest. Keywords crystallinity,pressurecooker,sorghum?ber,thermalproperties Macromol. Symp. 2020,391,1900129   Figure 3. TGA curves of untreated and treated sorghum in the tempera- turerangeof,a)40–700°C,b)100–300°C. [1] C. Borsoi,

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