APPLICATION OF COFFEE PEEL WASTE AS RAW MATERIAL FOR XYLOOLIGOSACCHARIDE PRODUCTION

Anak Agung Istri Ratnadewi[1], Handariatul Masruroh[2],
Suwardiyanto[3], Agung Budi Santoso[4]

ABSTRACT: Coffee is the second most common trade commodity in the world after petroleum. The coffee industry generates large amounts of waste in the form of coffee peels. Coffee peel waste consist of lignocellulose containing hemicellulose and other chemical compounds. The objective of this research was to extract xylan, the main component of hemicellulose from coffee waste, and to utilize the xylan in the production of xylooligosaccharide (XOS). Xylan was extracted from coffee waste using NaOH solution and neutralized by HCl 6 M. Afterward, xylan was precipitated using ethanol. Various NaOH concentrations (4, 8, 12 and 16 % w/v) were used to obtain the xylan. NaOH solution with a concentration of 12 % results 43 % extraction of xylan from coffee waste. Xylan obtained from the extraction was hydrolyzed using endo-β-1,4-xylanase from Bacillus sp. to produce XOS. The incubation time of enzyme-substrate was observed at 40 ºC, pH 5 and enzyme dose of 23.6 U. Thin layer chromatography results showed that the hydrolyzed products of xylan are XOS with composition xylobiose (X2), xylotriose (X3), xylotetraose (X4) and xylopentaose (X5). LC-MS studies revealed that X2 is the dominant product with the concentration of 6.00 ppm. This research demonstrates the potential to utilizate coffee peel waste as a source of xylan for the production of XOS.

Index terms: Coffee peel waste, endo-β-1,4-xylanase, xylooligosaccharide (XOS).

1 INTRODUCTION

In the last decade, there has been an increase in the utilization of bioactive compounds derived from plant as a substitute for the synthetic molecules used in the food and pharmaceutical as a guard of gastrointestinal health. Consumer awareness on healthy food, alternative pharmaceuticals and prohibitions of the use of antibiotics as growth promoter in the feed of poultry and livestock, phobia against excessive antibiotics and increase of the drug cost are some of the drivers of the research on the bioactive compound derived from plant having potency in guarding the intestinal health called probiotics (Samanta, et al., 2013). Prebiotics are a selected fermentation composition that produces a particular change in the composition and/or activity of gastrointestinal tract microbiota which leads to the health benefits for the host (Roberfroid et al., 2010). Some of the indigestible food materials appear as the candidates of prebiotics including galactans, fructan, XOS and β-glucan or arabinoxylan and can be produced from lignocellulose biomass which is abundant and renewable (Cummings et al., 2001; Mei et al., 2011,Samanta, et al., 2012; Achary et al., 2011; Saha 2003). XOS from corn stover and garlic straw have been reported having prebiotics effects by inducing the production of lactate, formate, and SCFA (acetate, propionate and butyrate) and increasing the population of Bifidobacterium and Lactobacillus bacteria beneficial for the gastrointestinal health (Buruiana et al., 2016; Kallel et al., 2014). The rise in demand for prebiotics precipitates this research into XOS production.

XOS are sugar oligomers composed of some xylose monomers with a degree of polymerization between 2 – 10. Production of XOS has received attention because it has potential to be applied in the production of food containing prebiotics. XOS has prebiotic effects when it is consumed as a part of the food. XOS with a low degree of polymerization (DP) have been proven to promote proliferation of bifidobacteria, beneficial microorganisms in human intestine (Brienzo, 2010). XOS is non digestible food ingredient and gives a positive effect by stimulating the growth of beneficial bacteria within the colon (Achary et al., 2011) XOS can be produced from waste lignocellulose containing xylan (Brienzo et al., 2010). Generally, xylan is in the complex of xylan-lignin within biomass containing lignocellulose. Because hydrolysing xylan is difficult of the difficulty of XOS production has to be conducted in two steps. First xylan must be extract from biomass containing lignocellulose. Then, it must be extract be hydrolysed enzymatically or chemically. XOS production through enzymatic xylan hydrolysis is preferable since it does not

[1]Center for Development of Advanced Science and Technology - Universitas of Jember - Jalan Kalimantan 37 - Jember 68121 Indonesia - istri_dewi.fmipa@unej.ac.id

[2,3,4]Departement of Chemistry - Faculty of Mathematics and Natural Sciences - Universitas of Jember, Jalan Kalimantan 37, Jember 68121 - Indonesia - antok.fmipa@unej.ac.id, agungbs.fmipa@unej.ac.id, ndahanda09@gmail.com

result in a negative by-product (Aarchary & Prapulla, 2011). Lignocellulose biomass that has been reported to contain xylan for XOS production include corn cobs, natural grass, tobacco stalk, sugarcane bagasse, pigeon pea, and cotton stalks. (Aarchary & Prapulla, 2009; Akpinar et al., 2010; Brienzo et al.,2010; Samanta et al., 2012; Samanta et al., 2013). This study is evaluating a new biomass resource, i.e. coffee bean peel, as a raw material for xylan extraction.

Coffee as the second most traded word commodity after petroleum. It is produced in about 60 tropical and subtropical countries and exported as an agricultural product (Lashermes et al., 2008; Viera, 2008). It is also a popular beverage and its consumption has increased over last the 150 years (Daglia et al., 2000). While meeting increase demand, the growing coffee industry is also creating an environmental problem (Solange et al., 2011). Waste from the coffee industry includes coffee pulp (CP), coffee husk (CH), coffee silver peel (CSS) and spent coffee grounds (SCG). Coffee peel waste is obtained by separating coffee beans from the fruit. Spent coffee grounds are the waste from the process of dissolving grounded coffee with hot water in the instant coffee industry (Solange et al., 2011). Many types of research have been pursued about ways to add value of the waste of the coffee industry. Most of the research focuses on the utilization of SCG as a resource in the production of biodiesel, fuel pellets, ethanol, adsorbents, and antioxidants etc. (Kondamudi et al., 2008; Franca et al., 2009; Ramalakshmi et al., 2009). The other research on CP, CH and CSS try to use them as substrate in the production of xylanase, fungi, gibberellins (plant hormones), biogas etc. (Murthy & Naidu, 2012; Fan et al., 2001; Machado et al., 2002; Jayachandra et al., 2011). Each waste component has hemicellulose content of 2.3 % (CP), 7.0 % (CH) and 13.1 % (CSS) (Musatto et al., 2011; Franca et al., 2009; Murthy & Naidu, 2010). Since it is not economic to separate the components of coffee waste, hence this research exploits all the part of the coffee waste. Several base solutions were used to optimize the xylan extraction. Xylan extracted was hydrolyzed using endo-β-1,4-D-xylanase isolated from Bacillus sp. of the termite abdomen system to produce XOS (Ratnadewi et al., 2007)

2 MATERIALS AND METHODS

Materials

XOS standards (X2, X3, X4, and X5) are products of Megazyme, Ireland. TLC plate Silica Gel 60 F254 was from Merck, Germany. Coffee peel waste was robusta varieties of local milling in the Silo district, Jember Regency. Coffee peel was ground to 100 mesh then dried at 60 °C until constant weight. Material was stored in a dry place until needed.

Microorganisms

Bacillus sp. from termite abdominal system was used to produce endo-β-1,4-xylanase which was kept at – 20 °C in glycerol stock.

Delignification of coffee peel waste

Coffee peel (10 g) was soaked in 100 mL of 0.5 % NaOCl solution for 5 h at 28 °C and rinsed with water afterward. The delignified sample was subjected to the xylan extraction process.

Xylan extraction from delignified coffee peel waste

A solid sample with moisture content of 9.56 ± 0.19 % (w/w) from the delignification process was soaked in NaOH solution for 24 h at 28 °C with a biomass : solution ratio of 1: 10 (w/v). The concentrations of NaOH were 2, 4, 8 and 12 % (w/v) The mixture was centrifuged at 10,000 rpm for 20 min. The supernatant was separated and neutralized with drops of HCl 6 N. Centrifugation was applied to the neutral sample at 10,000 rpm for 10 min and the supernatant of this process was mixed with ethanol 95 % (w/w) with volume ratio 1 : 3 (supernatant : ethanol). The mixture was centrifuged at 10,000 rpm for 10 min and the precipitate was collected and dried. Crude xylan content was calculated using the equation below:

$$\text{TrueYield Crude xylan (\%)} = \frac{\textit{final weight}}{\textit{initial weight}} \times 100\%$$

Xylan characterization by Fourier Transform Infrared Spectroscopy (FT-IR)

Xylan extracted from coffee peel waste was characterized using Fourier Transform Infrared Spectroscopy (FT-IR) HTS-XT, Bucket Optics GBH, Ettlinger at 400 – 4000 cm^{-1}. The sample of 10 mg in powder form was used in this characterization.

Enzymatic hydrolysis of xylan from coffee peel waste using endo-β-1,4-xylanase

Endo-β-1,4-xylanase with specific activity of 23.60 U/mg and total protein content of 0.454 mg. 125 µL crude enzyme was added to 125 µL xylan substrate 1 % (w/v, dry base) extracted from coffee peel waste. The mixture was incubated at 40 °C for 16, 24, 32 and 48 h. Total reducing

sugar was determined using DNS reagent and the absorbance was measured at 550 nm (G. Miller, 1959)

Qualitative analysis of hydrolysis products using thin layer chromatography (TLC)

XOS products from the enzymatic hydrolysis process were separated by means thin layer chromatography (TLC). The sample was dropped onto a silica plate and elution was done using the mixture of 1-butanol: acetic acid: water with a ratio 2: 1: 1 (v/v). XOS was detected by spraying solution of naphthol and H_2SO_4 in ethanol followed by drying at 100 °C.

Quantitative analysis of hydrolysis products by LC-MS

Enzymatic hydrolysis products of xylan were quantified using Liquid Chromatography-Mass Spectrometry (LC-MS) equipped with a Waters Sugar Pak I column (6.5 x 300 mm) at 80 °C and a mobile phase flow rate of 0.5 mL m^{-1} with water as the solvent. 10 µL of the sample was injected manually. XOS concentration was calculated by comparing the peak area of the sample and standard. The standards were xylose, xylobiose, xylotetraose, xylopentaose, and xylohexaose.

3 RESULTS AND DISCUSSION

True yield Xylan

Coffee peel waste is an abundant lignocellulosic waste and a potential source for xylan extraction. Recently there is an increase in the study of xylan extraction since xylan can be converted to prebiotic which is beneficial as antiulcer, antitussive and immunostimulant (Ebringerova et al., 2002; Akpinar et al., 2007). Xylan is the major component of hemicellulose within plant cell walls and which has covalent bonds between lignin and cellulose which form lignocellulose complex (Gowdhaman & Ponnusani, 2015). Xylan within lignocellulosic biomass can be extracted using several solvents such as alkaline solution, organic solvents, dilute acidic solution, enzyme, and water. Extraction by alkaline solution is prefere because hemicellulose by hydrolyzing the ester bond, which cause swelling of the cellulose and decrease the crystallinity of the cellulose (Samanta et al., 2013). It was reported that hydroxyl ion provokes the swelling of cellulose and destruction of the hydrogen bond between cellulose and hemicellulose eventually

xylan can be extracted easily (Al-Sheraji et al., 2013). Samanta et al. (2013) investigated the xylan extraction of pigeon pea using two alkaline solutions, i.e. NaOH and KOH and no difference among them. Based on this result, NaOH solution was used to extract xylan from coffee peel waste by soaking at room temperature for 24 h followed by neutralization using HCl 6 M and precipitation of xylan polymer using ethanol. The concentrations of NaOH solution were 4, 8, 12 and 16 % (w/v) to optimize the insoluble xylan. The result of insoluble xylan is tabulated in Table 1.

In the Table 1 is present that insoluble xylan recovery efficiency increased with increasing NaOH concentration from 4 to 12 % and decrease afterward. The more NaOH used for extraction lthe more xylan could be extracted up until 12%. Further increases in NaOH concentration causes xylan degradation, eventually lowering the xylan yield (Da Silva et al., 2012). It can be concluded that NaOH solution of 12 % is the optimum concentration for extracting of insoluble xylan from coffee peel waste with the yield of 9.73 %. This concentration has a relative yield of 43 % (Fig. 1) where relative yield was calculated based on hemicellulose content of coffee pulp, coffee husk and coffee silver peel (Murthy & Naidu, 2012).
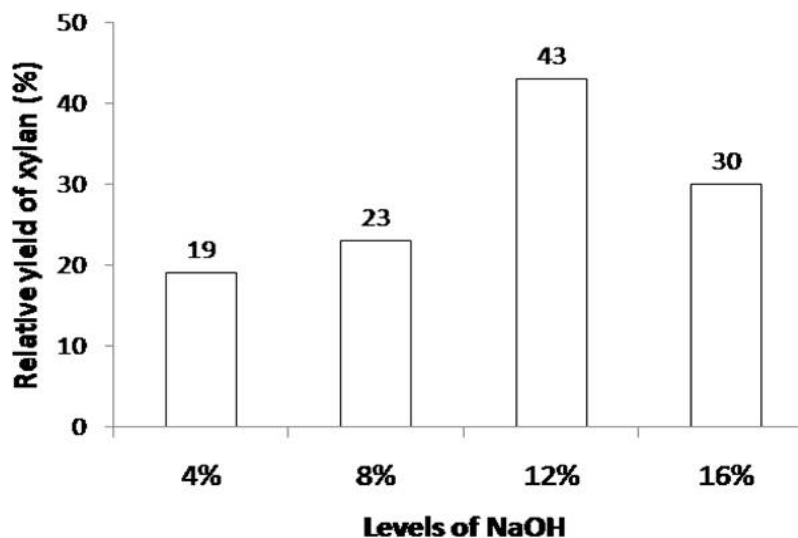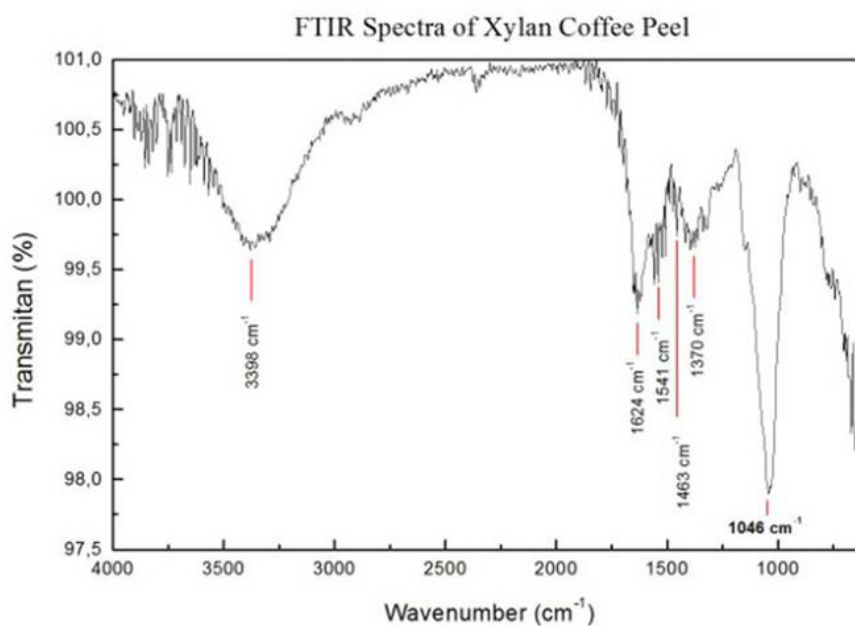
FTIR Spectra

Infrared spectroscopy is used to identify functional groups of the organic compound. IR spectra show a specific signal of the functional group. This technique was employed to identify functional groups of xylan extracted from coffee peel waste using alkaline solution.

Fig. 2 is the IR spectra of xylan from coffee peel waste showing the characteristic of xylan. The spectra show vibrations at the wavenumber of 3398, 1642, 1541, 1463, 1370, 1048 and 652.29 cm^{-1}. These vibrations show the pattern of xylan IR spectra (Kumar & Negi, 2012). The broadband of 3398 cm^{-1} is from the vibration of the hydroxyl group of xylan (Oliviera et al., 2010). The absorbed water within xylan peaked at 1642 cm^{-1} and aromatic C=C bond appeared at 1541 cm^{-1} (Zhou et al., 2011). The presence of lignin vibration in these spectra infers that the delignification process was not complete (Kumar & Negi, 2012). The vibration of methoxy groups (O-CH3) appears at the wavenumber of 1463 cm^{-1}, this band may be due to the possible existence of 4-0-methyl-glucuronosyl within the extracted xylan (Yang et al., 2007).

TABLE 1 - Effect of alkali concentration on the recovery of insoluble xylan from coffee peel waste.

Concentration of NaOH (w/v)	Recovery of insoluble xylan (% of raw materials) *
4 %	4,33±0,11
8 %	5,13±0,31
12 %	9,73±0,11
16 %	6,67±0,23

FIGURE 1 - Effect of NaOH concentration on relative yield of xylan from coffee peel waste.

FIGURE 2 - FT-IR spectra of xylan from obtained through 12 % sodium hydroxide application from coffee peel waste

The peak at 1370 cm^{-1} is the bending mode of C-H and C-O of xylan (Peng et al., 2010). A sharp absorbance at 1048 cm^{-1} characterizeds xylan vibration due to the stretching mode of C-C, C-O and bending mode of C-OH within xylan (Colom et al., 2003).

XOS production from xylan of coffee peel waste

Xylan extracted from coffee peel waste was hydrolysed to produce XOS. Xylan is a heteropolysaccharide. Xylose is the component of the main chains of xylan and is connected by β-1,4-glycosidic bonds with O-acetyl, arabinosyl, and methyl-glucuronosyl side chains (Abirami et al., 2011). Xylan can be employed in the production of XOS through enzymatic hydrolysis using endo-β-1,4-D-xylanase where the enzyme cuts the chain within xylan randomly. XOS is a carbohydrate oligomer with xylose as the monomeric unit and has prebiotic properties. The potential for XOS production from lignocellulosic waste has positive economic prospects, as the demand for prebiotics is increasing in food and pharmaceutical sectors (Goldman, 2009). XOS in this research is produced through hydrolysis of xylan from coffee peel waste by endo-β-1,4-D-xylanase from Bacillus sp. of termite abdominal system (Ratnadewi et al., 2007). The enzyme can hydrolyze oat xylan to produce XOS with the component such as xylobiose, xylotriose, xylotetraose and xylopentaose (Ratnadewi et al., 2016). XOS production in this research was

conducted at 40 ^{o}C, pH 5 and an enzyme dose 23.6 U. Incubation time was varied for 16, 24, 32 and 48 h and the effects of each respective incubation was observed. Specifically the observation measured the total sugar reduction. The data is presented in Fig. 3.

Researchers observed the greatest amount of total reducing sugar between 16-24 h at which point total reducing sugar decreased. It can be inferred that 24 h is the optimum time to hydrolyze xylan into XOS with total reduce sugar measured at 0.334 mg/ml. The decrease of total reducing sugar after 24 h was hypothesized to have caused by decrease of activity of endo-β-1,4-D-xylanase.

Thin layer chromatography was applied to analyze the component of XOS as the product of hydrolysis. The analysis results in separation as shown in Fig. 4. It shows the occurrence of some spot on the hydrolysis products (Line 4 & 6) which is around or parallels with the XOS standards i.e. xylobiose, xylotriose, xylotetraose and xylopentaose (Line 2) and the absence within the area of xylose. Based on this result, the primary components of the XOS are xylobiose (X2), xylotriose (X3), xylotetraose (X4) and xylopentaose (X5) without xylose. Other research has reported that XOS with DP 2 - 3 from garlic straw xylan and XOS with DP 2 – 6 from corn cob have potency as prebiotics (Kallel et al., 2014; Moura et al., 2007). It can be concluded that XOS hydrolysis product of xylan from coffee peel waste by endo-β-1,4-D-xylanase may also have application as probiotics.
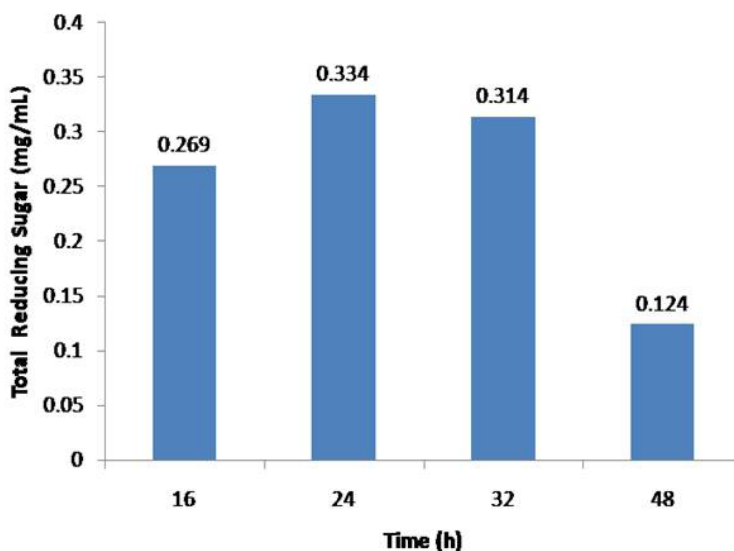

FIGURE 3 - Time course for production XOS generated from xylan coffee peel waste (1 %) using with 23.6 U/ mL of endo-β-1,4-xylanase from *Bacillus* sp. of termite abdominal system at pH 5 and 50 ^{o}C .

C-MS was used for quantitative analysis of each XOS composition arising from xylan hydrolysis. The results of the analysis are presented in Figure 5 and 6. The amount of XOS was determined by comparing the peak area of the sample and the standard. The concentrations of X2, X4, and X6 in the sample are 6.00, 0.802 and 0.832 ppm respectively. It shows that X2 is the dominant product of xylan hydrolysis by endo-β-1,4-D-xylanase. Aachary dan Prapulla (2011) have reported that XOS with DP 2-3 has physiological benefit for human and animals, thus the hydrolysis products of xylan from coffee peel waste may also be used as a substrate for prebiotic productions.
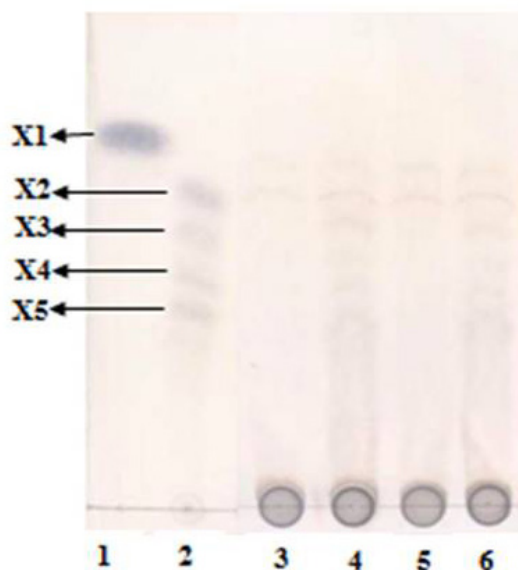

FIGURE 4 - Thin layer chromatogram of component of XOS as the product of enzymatic hydrolysate of xylan from coffee peel waste. Lane 1, xylose (1 μg/μl); 2 XOS standard mixture (1 μg/μl); 3 & 5 control (using inactive enzyme); 4 & 6 XOS generated by 23,6 U of respectivly at 40 ℃, pH 5 with 24 h of incubation.
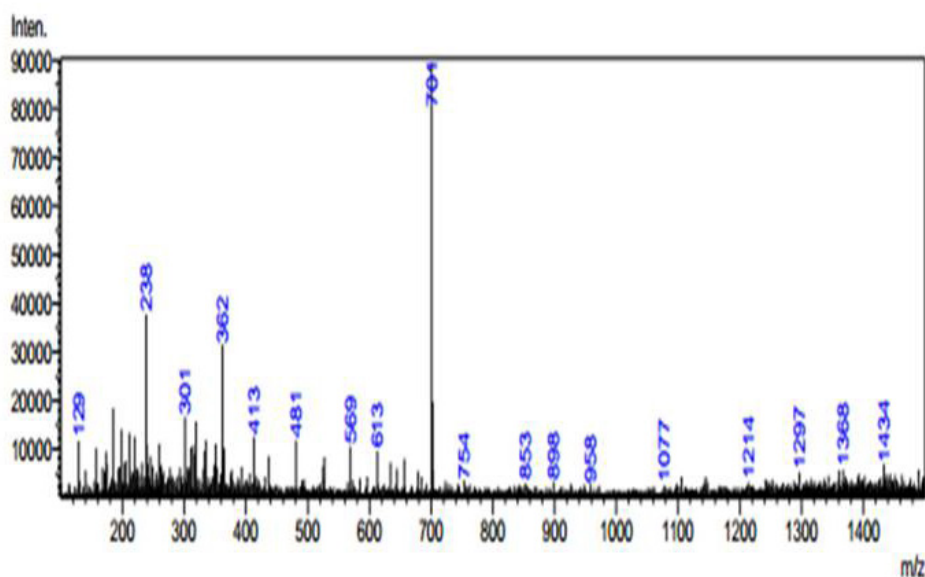

FIGURE 5 - Mass spectra of XOS of enzymatic hydrolysate of xylan from coffee peel wast.
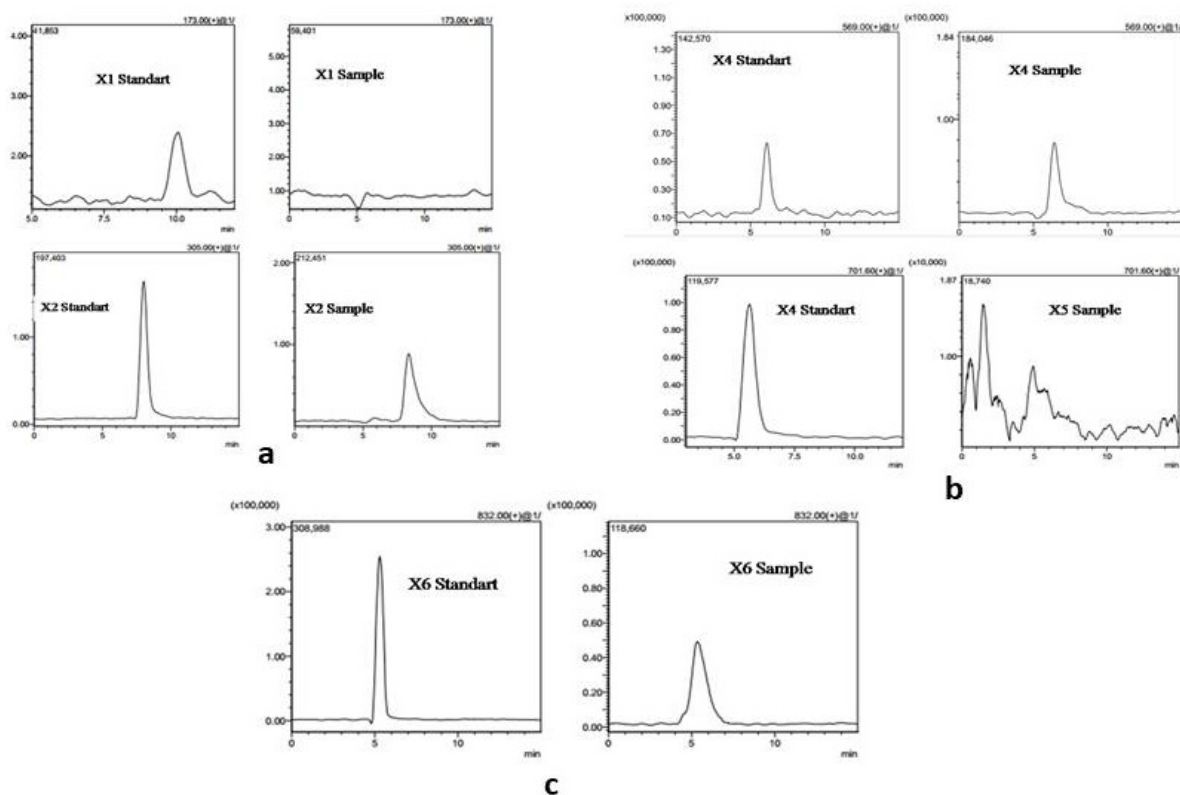
FIGURE 6 - Chromatogram LC-MS of XOS of enzymatic hydrolysate of xylan from coffee peel waste.

4 CONCLUSIONS

This researc identified coffee peel as a source of xylan for XOS production. The more NaOH used for extraction lthe more xylan could be extracted up until 12% with the yield of 43% relative to original hemicellulose content of the feedstock. Utilization of endo-β-1,4-D-xylanase from *Bacillus* sp. for xylan hydrolysis results in XOS with the degree of polymerization 2-5 i.e. xylobiose, xylotriose, xylotetraose, xylopentaose, and xylohexaose. The LC-MS analysis shows that X2 is the dominant product with a concentration of 6.0 ppm. Though produced concentrations of XOS measured less than would be produced when using the method employed in this study is cheaper and documents a novel substrate for XOS production. Further, it demonstrated that coffee peel waste from the agricultural sector may be uses in production of XOS rather than discarded.

6 REFERENCE

AACHARY, A. A.; PRAPULLA, S. G. Value addition to corn cob: production and characterization of xylooligosaccharides from alkali pretreated ligninsaccharide complex using *Aspergillus oryza* MTCC5154. **Bioresource Technology**, 100, 991–995. 2009.

AACHARY, A. A.; PRAPULLA, S. G. Xylooligosaccharides (XOS) as an emerging prebiotic: microbial synthesis, utilization, structural characterization, bioactive properties and applications. **Comprehensive Reviews in Food Science and Food Safety**, 10, 2–16, 2011.

ABIRAMI, V.; MEENAKHSI, S.A.; KANTHYMATHY K.; BARHATHIDASAN R.; MAHALINGAM R.; PANNEERSELVAM A. Partial purification and characterization of anextracellular xylanase from *Penicillium janthinellum* and *Neurospora crassa*. **J. Natural Product Plant Resources** 1(4), 117-125.2011.

AKPINAR, O., AK, O.;KAVAS, A.; BAKIR, U.; YILMAS, L. Enzymatic production of xylooligosaccharides from cotton stalks. **Journal Agricultural and Food Chemistry**,55, 5544-5551,2007.

AKPINAR, O.; ERDOGAN, K.; BAKIR, U.; YILMAZ, L. Comparison of acid and enzymatic hydrolysis of tobacco stalk xylan for preparation of xylooligosaccharide. **LWT-Food Science and Technology**, 43, 119–125. 2010

AL-SHERAJI, S. H.; ISMAIL, A.; MANAP, M. H.; MUSTAFA, S.; YUSOF, R. M.; HASSAN, F. A. Prebiotic functional foods: A Review. **Journal of Functional Foods, 5(4),** 1542-1553, 2013.

BRIENZO, M.; CARVALHO, W.; MILAGRES, A. M. F. Xylooligosaccharides production fromalkali pretreated sugarcane bagasse using xylanase from Thermoascus aurantiacus. **Applied Biochemistryand Biotechnology**, 162, 1195–1205, 2010

BURUIANA, C. T.; GÓMES, B.; VIZIREANU, C.; GARROTE, G. Manufacture and evaluation of xylooligosaccharides from corn stover as emerging prebiotic candidates for human health. **LWT-Food Science and Technology**, 77, 449-459, 2016.

COLOM, X.; CARRILLO, F.; NOGUES, F.; GARRIGA, P. Structural analysis of photodegraded wood by means of FTIR spectroscopy. **Polymer Degradation and Stability**, 80,543-549, 2003

CUMMINGS, J.; MACFARLANE, G.; ENGLYST, H. Prebiotic digestion and fermentation. American Journal of Clinical Nutrition, 73(2), 415S-420S, 200.

DAGLIA, M.; PAPETTI, A.; GREGOTTI, C.; BERTE, F.; GAZZANI, G. In vitro antioxidant and ex vivo protective activities of green and roasted coffee. **Journal of AgriculturalandFood Chemistry,** 48, 1449–1454, 2000.

DA SILVA, A. E.; MARCELINO, H. R.; GOMES, M. C. S.; OLIVIERA, E. E.; NAGHASIMA, T.; EGITO, E. S. T. Xylan, a promising hemicellulose for pharmaceutical use. **Products and Application of Biopolymers**, 61-84, 2012.

EBRINGEROVA, A.; HROMADKOVA, Z.; MALOVIKOVA, A.; HRIBALOVA, V. Immunomodulatory activity of acidic xylans in relation to their structural and molecular properties. **International Journal of Biological Macromolecules**, 30, 1–6, 2002

FAN, L.; PANDEY, A.; SOCCOL, C. R. *Flammulina velutipes* on coffee husk and coffee spentground. **Brazilian Archives of Biology and Technology**,44, 205–212, 2001.

FRANCA, A. S.; OLIVEIRA, L. S.; FERREIRA, M. E. Kinetics and equilibrium studies of methylene blue adsorption by spent coffee grounds. **Desalination 249**, 267–272, 2009.

GOLDMAN, N. Methods for optimizing enzymatic hydrolysis of xylan to improve xylooligosaccharides yields. **Basic Biotechnology**, 5, 31-36, 2009.

GOWDHAMAN, D.; PONNUSANI, V. Production and optimization of xylooligosaccharides from corncob by *bacillus aerophilus* KGJ2 xylanase and its antioxidant potential. **International Journal of Biological Macromolecules**, 79, 595–60, 2015.

JAYACHANDRA, T.; VENUGOPAL, C.; ANU APPAIAH, K. A. Utilization of phytotoxic agro waste-coffee cherry husk through pretreatment by the ascomycetes fungi Mycotypha for biomethanation. **Energy for Sustainable Development**, 15(10), 104-108, 2011.

KALLEL, F.; DRISS, D.; BOUAZIZ, F.; NEIFER, M.; GHORBEL, R.; CHAABOUNI, S, E. Production of xylooligosaccharides from garlic straw xylan by purified xylanase from *Bacillus mojavensis ueb-fk* and their *in vitro* evaluations prebiotics. **Food and Bioproduct Processing**, 94, 536-546, 2015.

KONDAMUDI, N.; MOHAPATRA, S. K.; MISRA, M. Spent coffee grounds as a versatile source of green energy. **Journal of Agricultural and Food Chemistry,** 56, 11757–11760, 2008.

KUMAR, S.; NEGI, Y. S. Corn cob xylan-based nanoparticle: Ester product of 5-aminosalicylic acid for possible targeted delivery of a drug. **Journal of Pharmaceutical Sciences and Research**, 4(12), 1995-2003, 2012.

LASHERMES, P.; ANDRADE, A. C.; ETIENNE, H. Genomics of coffee, one of the world's largest traded commodities, 2008.

MACHADO, C. M. M.; SOCCOL, C. R.; OLIVEIRA, B. H.; PANDEY, A. Gibberellic acid production by solid-state fermentation in a coffee husk. **Applied Biochemistry and Biotechnology**, 15, 102–106, 2002

MEI, G.; CAREY, C.; TOSH, S.; KOSTRZYNSKA, M. Utilization of different types of dietary fiber by potential probiotics. Canadian Journal of Microbiology, 57(10), 857-865, 2011.

MOURA, P.; BARATA, R.; CARVALHEIRO, F.; GIRIO, F.; LOUREIRO, M. C.; ESTEVES, M. P. In vitro fermentation of xylo-oligosaccharides from corn cobs autohydrolysis by *Bifidobacterium* and *Lactobacillus* strains. **LWT**, 40, 963-972, 2007.

MURTHY, P.S.; NAIDU M. M. Protease production by *Aspergillus oryzae* in solid state fermentation utilizing coffee by-products. **World Applied Science Journal**, 8(2), 199–205, 2010.

MURTHY, P. S.; NAIDU M. M. Production and application of xylanase from *Penicillium sp.* utilizing coffee by-products. **Food Bioprocess Technology**, 5, 657-66, 2012.

MUSATTO, S. I.; MACHADO, E. M. S.; MARTHIN, S.; TEIXEIRA, J. A. Production, composition, and application of coffee and its industrial residues. **Food Bioprocess Technology**, 4, 661-672, 2011.

OLIVIERA, E. E.; SILVA, A. E.; JUNIOR, T. N.; GOMES, M. C. S.; AGUIAR, M. A.; MARCELINO, H. R.; ARAUJO, I. B.; BAYER, M. P.; RICARDO, N. M. P. S.; OLIVIERA, A. S.; EGITO, E. S. T. Xylan from corn cobs, a promising polymer for drug delivery: Production and characterization. **Bioresource Technology**, 101, 5402-5406, 2010.

PENG, F.; REN, J. L.; XU, F.; BIAN, J.; PENG, P.; SUN, R. C. Fractional study of alkali-soluble hemicelluloses obtained by graded ethanol precipitation from sugar cane bagasse. **Journal of Agricultural and Food Chemistry**, Vol. 58: 1768-1776, 2010.

RAMALAKSHMI, K.; RAO, L. J. M.; TAKANO-ISHIKAWA, Y.; GOTO, M. Bioactivities of low-grade green coffee and spent coffee in different in vitro model systems. **Food Chemistry**, 115, 79–85, 2009.

RATNADEWI, A. A. I.; HANDAYANI W.; PUSPANINGSIH, N. N. T. Produksi dan karakterisasi, enzim *β*-endoxilanase dari bakteri sistem intestinal rayap. **Jurnal Ilmu Dasar** 8(2): 110, 2007.

RATNADEWI, A.A.I; KURNIAWAN, Andika Ade; HANDAYANI, Wuryanti. Optimization pH of Enzymatic Hydrolysis of Endo-1,4-β-Xylanase for Xylooligosaccharides Production. **UNEJ e-Proceeding**, [S.l.], feb. 2016. ISSN 2686-0783. Available at: <https://jurnal.unej.ac.id/index.php/prosiding/article/view/2478

ROBERFROID, M.; GIBSON, G.; HOYLES, L.;MCCARTENEY, A.; RASTALL, R.; ROWLAND, I.; WOLVERS, D.; WATZL, B.; SZAJEWSKA, H.; STAHL, B.; GUARNER, F.; RESPINDEK, F.; WHELAN, K.; COXAM, V.; DAVICCO, M.; LÉOTOING, L.; WITTRANT, Y.; DELZENNE, N.; CANI, P.; NEYRINCK, A.; MEHEUST, A. Prebiotic effects: metabolic and health benefits. **British Journal of Nutrition**, 104(S2), S1-S63, 2010.

SAHA, B. C. Hemicelluloses bioconversion. **Journal of Industrial Microbiology and Biotechnology**, 30, 279–291, 2003

SAMANTA, A. K.; SENANI, S. S.; KOLTE, A. P.; SRIDHAR, M.; SAMPATH, K. T.; JAYAPAL, N. Production and in vitro evaluation of xylooligosaccharides generated from corn cobs. **Food Bioproducts Processing**, 90, 466–474, 2012.

SAMANTA, A. K.; JAYAPAL, N.; KOLTE, A. P.; SENANI, S.; SRIDHAR, M.; MISHRA, S.; PRASAD, C. S.; SURESH, K. P. Application of pigeon pea (*Cajanus cajan*) stalks as raw material for xylooligosaccharides production. **Application Biochemistry Biotechnology** 169, 2392-2404, 2013.

SOLANGE I.; MUSSATTO; ERCÍLIA M. S; MACHADO; SILVIA MARTINS; JOSÉ A. Teixeira. Production, Composition, and Application of Coffee and Its Industrial Residues. **Food Bioprocess Technol**, 4, 661-672, 2011.

VIEIRA, H. D. 2008. Coffee: The plant and its cultivation. **Plant-Parasitic Nematodes of Coffee**, 1, 3-18.

YANG, H.; YAN, R.; CHEN, H.; LEE, D. H.; ZHENG, C. Characteristics of hemicellulose, cellulose and lignin pyrolysis. **Fuel**, 86, 1781-1788, 2017.

ZHOU, G.; TAYLOR, G.; POLLE, A. FTIR-ATR-based prediction and modeling of lignin and energy contents reveal independent intra-specific variation of these traits in bioenergy poplars. **Plant Methods**, 7, 1-9, 2011.