ภาคผนวก ก
ผลการบำบัดสารเมโธมิล

ตารางที่ ก.1 ผลของการศึกษาชนิดของแอโรเจลในการบำบัดสารเมโธมิลร่วมกับสารเปอร์ซัลเฟต

Time	**PS Only**		**PS +Aerogel**		**PS + Sulfide-modified zero-valent zinc aerogel**		**PS + Sulfide-modified aerogel**		**Aerogel Only**	
	Con.	**Ct/C0**	**Con.**	**Ct/C0**	**Con.**	**Ct/C0**	**Con.**	**Ct/C0**	**Con.**	**Ct/C0**
0	4.850	1.000	4.673	1.000	4.929	1.000	5.057	1.000	5.541	1.000
5	4.137	0.853	3.709	0.794	4.047	0.821	4.819	0.953	4.253	0.767
10	3.980	0.821	3.511	0.751	3.952	0.802	4.596	0.909	4.124	0.744
20	3.896	0.803	3.379	0.723	3.831	0.777	4.374	0.865	4.070	0.734
30	3.676	0.758	2.917	0.624	3.075	0.624	3.859	0.763	3.900	0.704
60	3.377	0.696	2.751	0.589	2.899	0.588	3.302	0.653	4.070	0.734
90	3.250	0.670	2.615	0.560	2.744	0.557	2.804	0.554	4.368	0.788
120	2.869	0.592	2.494	0.534	2.538	0.515	2.360	0.467	4.409	0.796

ตารางที่ ก.2 ผลของการศึกษาความเข้มข้นของสารเปอร์ซัลเฟตในการบำบัดสารเมโธมิลร่วมกับสารเปอร์ซัลเฟต

Time	**PS 200 mg/L**		**PS 400 mg/L**		**PS 500 mg/L**		**PS 600 mg/L**	
	Con.	**Ct/C0**	**Con.**	**Ct/C0**	**Con.**	**Ct/C0**	**Con.**	**Ct/C0**
0	4.670	1.000	4.880	1.000	4.642	1.000	4.948	1.000
5	3.974	0.852	4.846	0.994	4.385	0.945	4.743	0.962
10	3.865	0.828	4.713	0.966	4.301	0.927	4.586	0.930
20	3.912	0.838	4.340	0.891	4.106	0.885	4.560	0.923
30	4.121	0.883	4.173	0.858	3.912	0.842	4.143	0.839
60	4.059	0.870	3.109	0.641	3.664	0.789	3.674	0.744
90	4.044	0.867	2.651	0.544	3.222	0.693	3.052	0.617
120	3.963	0.850	2.277	0.469	2.895	0.623	2.803	0.566

ตารางที่ ก.3 ผลของการศึกษาระยะเวลาในการสัมผัสในการบำบัดสารเมโธมิลร่วมกับสารเปอร์ซัลเฟต

เวลา (นาที)	**สารเมโธมิล**	
	Con.	**Ct/C0**
0	4.834	1.000
5	4.681	0.973
10	4.642	0.964
20	4.400	0.918
30	4.222	0.881
60	3.762	0.787
90	3.691	0.772
120	3.457	0.723
180	3.051	0.639
240	2.720	0.570
300	2.415	0.506
360	2.229	0.463
1440	1.954	0.407

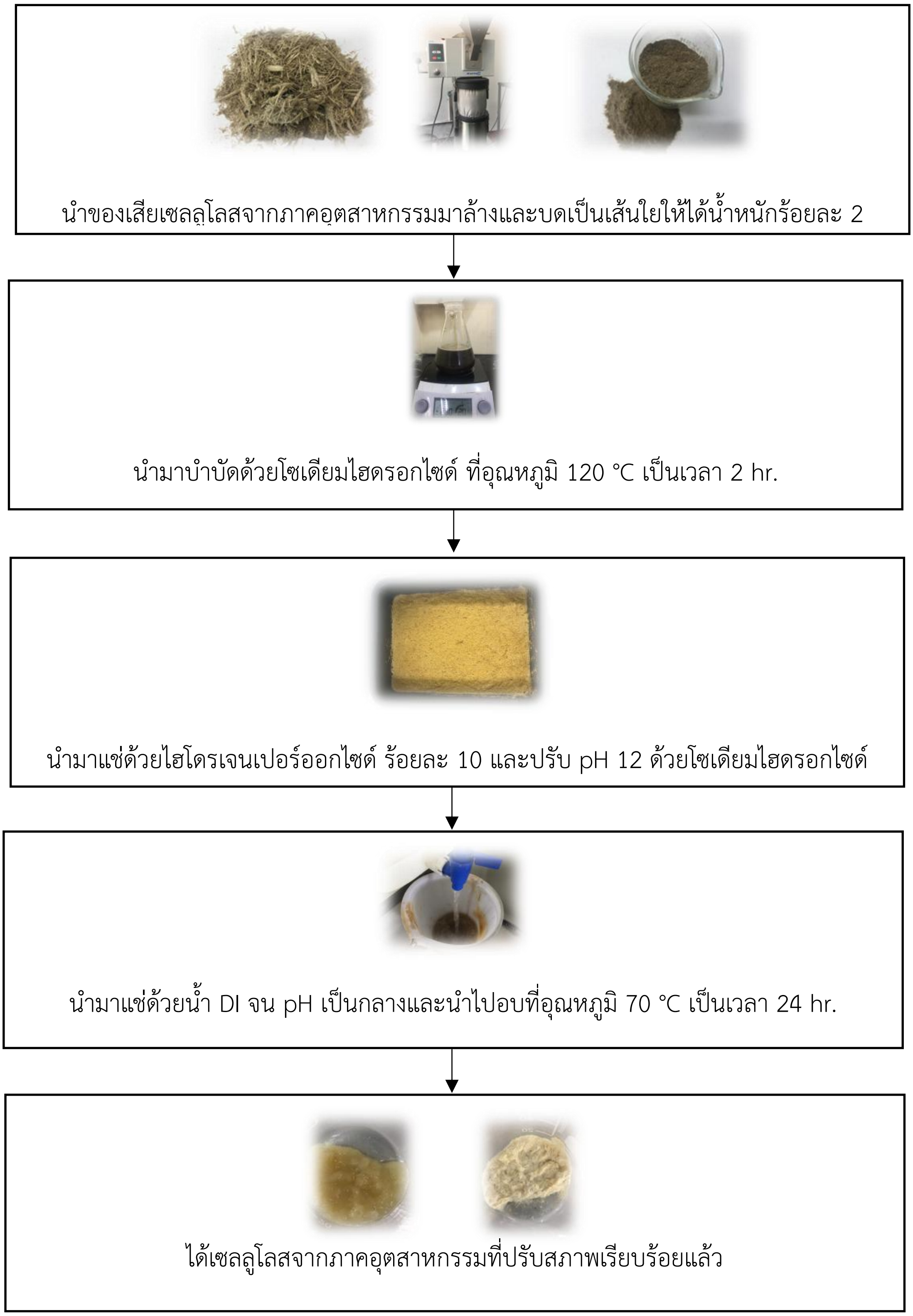
ตารางที่ ก.4 ผลของการศึกษาความเร็วรอบในการกวนในการบำบัดสารเมโธมิลร่วมกับสารเปอร์ซัลเฟต

เวลา (นาที)	**100 รอบต่อนาที**		**150 รอบต่อนาที**		**200 รอบต่อนาที**		**250 รอบต่อนาที**	
	Con.	**Ct/C0**	**Con.**	**Ct/C0**	**Con.**	**Ct/C0**	**Con.**	**Ct/C0**
0	4.710	1.000	4.965	1.000	5.019	1.000	4.972	1.000
5	4.033	0.857	4.688	0.945	4.562	0.910	4.873	0.981
10	3.839	0.832	4.472	0.900	4.073	0.812	4.011	0.807
20	3.914	0.818	3.731	0.752	3.517	0.701	4.057	0.816
30	3.571	0.759	3.460	0.697	3.165	0.630	4.195	0.843
60	3.304	0.703	3.217	0.648	2.848	0.567	3.357	0.675
90	2.835	0.603	3.117	0.628	2.375	0.473	2.913	0.586
120	2.462	0.523	2.784	0.561	2.191	0.438	2.931	0.589
180	2.278	0.485	2.788	0.562	2.103	0.420	2.558	0.515
240	2.067	0.440	2.326	0.470	1.835	0.367	2.220	0.447
300	1.997	0.425	2.403	0.484	1.699	0.339	2.003	0.404
360	2.018	0.429	2.261	0.456	1.744	0.349	1.787	0.360

ตารางที่ ก.5 ผลของการศึกษาค่าความเป็นกรดด่างในการบำบัดสารเมโธมิลร่วมกับสารเปอร์ซัลเฟต

เวลา (นาที)	**pH 3**		**pH 5**		**pH 7**		**pH 9**	
	Con.	**Ct/C0**	**Con.**	**Ct/C0**	**Con.**	**Ct/C0**	**Con.**	**Ct/C0**
0	4.888	1.000	4.412	1.000	4.796	1.000	4.709	1.000
5	5.353	1.097	6.114	1.386	4.687	0.977	4.660	0.979
10	5.581	1.144	5.769	1.308	4.581	0.955	4.416	0.896
20	4.825	0.988	5.534	1.254	4.538	0.947	4.076	0.835
30	4.535	0.932	4.891	1.109	4.412	0.920	4.053	0.886
60	3.328	0.686	4.154	0.940	4.170	0.870	3.963	0.796
90	3.215	0.661	4.663	1.054	4.078	0.851	3.563	0.716
120	2.904	0.594	4.248	0.961	3.931	0.820	3.359	0.711
180	3.166	0.651	3.793	0.858	2.988	0.624	3.115	0.613
240	3.484	0.713	3.633	0.826	2.807	0.585	2.705	0.535
300	2.993	0.614	3.409	0.773	2.700	0.563	2.491	0.522
360	2.902	0.595	3.315	0.752	2.634	0.549	2.353	0.477

ภาคผนวก ข
รูปประกอบการปรับสภาพของเสียชานอ้อยด้วยกระบวนการทางเคมี



นำของเสียเซลลูโลสจากภาคอุตสาหกรรมมาล้างและบดเป็นเส้นใยให้ได้น้ำหนักร้อยละ 2

นำมาบำบัดด้วยโซเดียมไฮดรอกไซด์ ที่อุณหภูมิ 120 °C เป็นเวลา 2 hr.

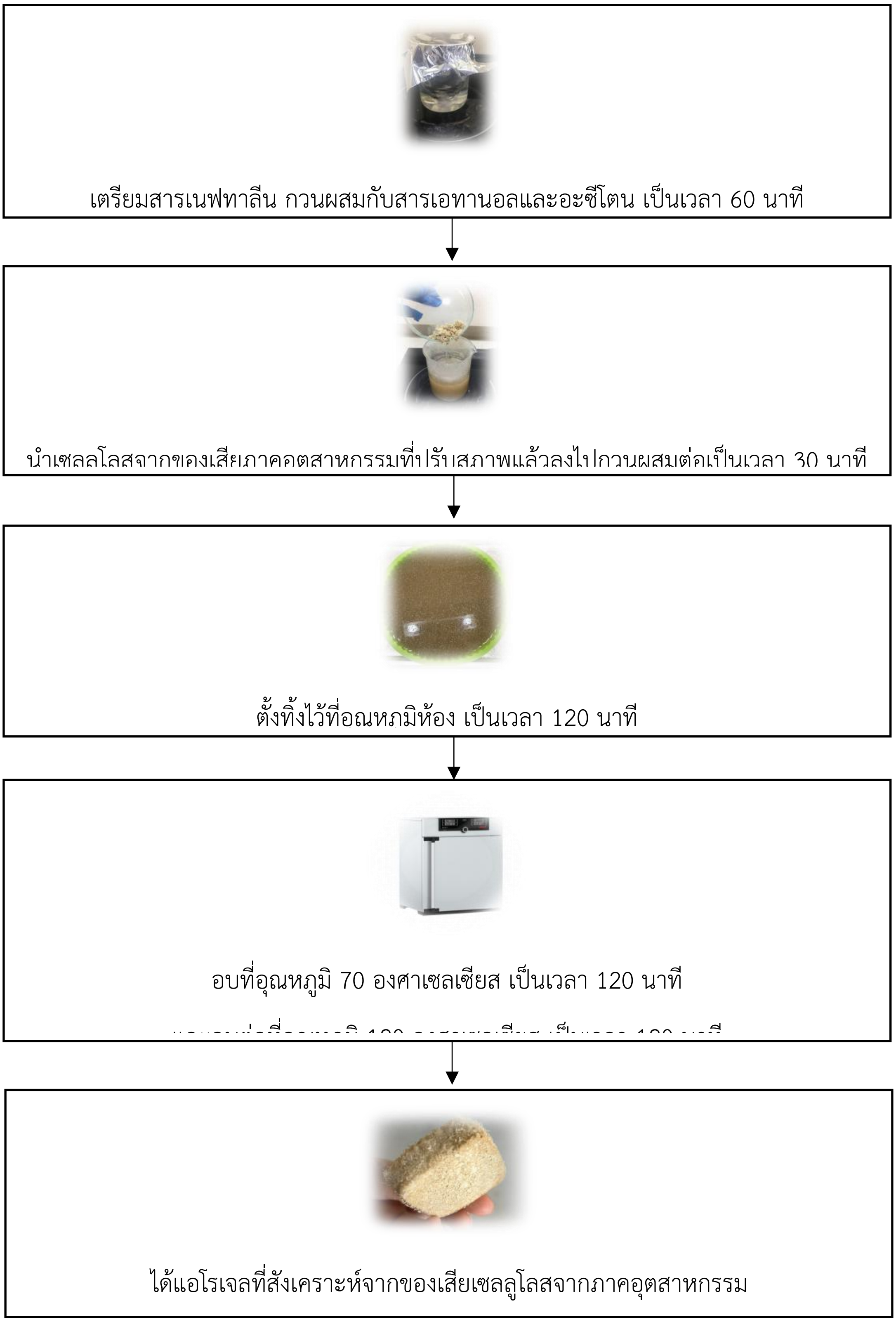
นำมาแช่ด้วยไฮโดรเจนเปอร์ออกไซด์ ร้อยละ 10 และปรับ pH 12 ด้วยโซเดียมไฮดรอกไซด์

นำมาแช่ด้วยน้ำ DI จน pH เป็นกลางและนำไปอบที่อุณหภูมิ 70 °C เป็นเวลา 24 hr.

ได้เซลลูโลสจากภาคอุตสาหกรรมที่ปรับสภาพเรียบร้อยแล้ว

รูปที่ ข.1 ขั้นตอนการปรับสภาพของเสียชานอ้อยด้วยกระบวนการทางเคมี

ภาคผนวก ค
รูปประกอบการสังเคราะห์แอโรเจล



รูปที่ ค.1 ขั้นตอนการสังเคราะห์แอโรเจลจากของเสียเซลลูโลส

ภาคผนวก ง

รูปประกอบการสังเคราะห์แอโรเจลผสมกับสังกะสีประจุศูนย์ที่ปรับปรุงด้วยซัลไฟด์ (S-ZV-Zn-Aerogel)

นำแอโรเจลที่สังเคราะห์ได้มาแช่ในสารละลาย $ZnCl_2$ 50 มิลลิลิตร เป็นเวลา 24 ชั่วโมง

ผสมสาร $NaBH_4$ 0.945 กรัม และ $Na_2S_2O_4$ 0.043 กรัม ในน้ำ DI 50 มิลลิลิตร

หยดสารละลาย 50 มิลลิลิตร ด้วยอัตราการไทเทรต 1 หยดต่อวินาที

กวนผสมเป็นเวลา 30 นาที และล้างด้วย Ethanol 3 ครั้ง

นำเข้าเครื่องอบที่อุณหภูมิ 70 องศาเซลเซียส เป็นเวลา 3 ชั่วโมง

จากนั้นก็จะได้ S-ZV-Zn-Aerogel ที่สังเคราะห์เสร็จเรียบร้อย

รูปที่ ง. ขั้นตอนการสังเคราะห์แอโรเจลที่ผสมกับสังกะสีประจุศูนย์ที่ปรับปรุงด้วยซัลไฟด์ (S-ZV-Zn-Aerogel)

ภาคผนวก จ
บทความทางวิชาการที่ได้รับการตีพิมพ์เผยแพร่ในระหว่างศึกษา

Characterization of aerogel from sugarcane bagasse

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Abstract :

Sugarcane is an important raw material in the sugar production industry. The sugar production process consists of seven main steps: harvesting, extraction, clarification, evaporation, sugar crystallization, crystal separation, and crystal drying. The extraction process involves grinding sugar cane to extract juice. The water is then collected in large containers for further processing. This is typically done using a grinder. However, when grinding sugarcane, bagasse waste is produced, which is a material left over from the production process. The sugar production industry produces a large amount of bagasse waste. Therefore, the researcher had an idea to synthesize this bagasse waste into an aerogel absorbent material from bagasse cellulose. The chemical composition of bagasse contains up to 45% alpha-cellulose. Therefore, bagasse is interesting to develop into cellulose aerogels. Aerogel refers to a synthetic material with low density and high surface area. This natural material can be used as a precursor for the synthesis of aerogels. In addition, the aerogel can be molded into the desired shape, which depends on the shape of the mold, and is left to become a gel with air by drying. The synthesis process of aerogel absorbent material using bagasse must be chemically treated with sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), and sulfuric acid (H_2SO_4) to remove hemicellulose and lignin. It can then be synthesized into cellulose aerogels. It uses an ambient pressure drying method to create aerogels. Ambient pressure drying is a simple and environmentally friendly technique. This research will use aerogels mixed with sulfide-modified zerovalent zinc aerogels to increase adsorption properties. The properties will be characterized using various techniques, including XRD, SEM, FTIR, XTM etc. From chemical pretreatment with sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), and sulfuric acid (H_2SO_4). Able to remove contaminants from bagasse waste and can reduce lignin and hemicellulose content, which is consistent with the results from the study of properties using FTIR, SEM, and XRD techniques. After that, the characteristics of the chemically treated bagasse were studied, and the bagasse was synthesized into an aerogel absorbent material mixed with sulfide-modified zerovalent zinc through an ambient pressure drying process. Later, the properties were studied using various techniques, namely SEM and XTM. The SEM technique found that the surface area characteristics of the aerogel were those of the mixed zerovalent zinc modified with sulfide. The fibers are not visible because the aerogel is coated with zerovalent zinc modified with sulfide. The results of the XTM technique revealed that when the aerogel was coated with zerovalent zinc modified with sulfide, the porosity decreased from 88% to 70%.

Keywords: Aerogel, Cellulose waste, Bagasse, The ambient pressure drying process, Zero-valent zinc

Introduction.

The industrial sector is an important force driving the domestic economy, including the food industry, automotive industry, electronic industry, rubber and plastic industry, paper industry, sugar industry, chemical industry, and petroleum industry. In the industrial sector, waste from the production process often occurs. For example, the sugar industry is a large agricultural product industry that can be processed in the sugar production process. Bagasse is a by-product remaining from the sugarcane grinding process by extracting sugarcane juice. Most of the remaining bagasse is sugarcane fiber. For 1 ton of fresh sugar cane, approximately 290 kilograms of bagasse will be left as waste from the production process. The sugar production industry in Thailand has a total of 57 sugar factories. In 2022, the Office of the Cane and Sugar Board reported that sugarcane consumption in the sugar production industry was approximately 94 million tons, which is considered high consumption [1]. As a result, the amount of bagasse waste remaining from the sugarcane crushing process is as much as 27 million tons, and some sugar production industries have not yet put bagasse to use or burned. However, the chemical composition of bagasse consists of approximately 45% alpha cellulose, 27% hemicellulose, 21% lignin, and 7% others. Therefore, the researcher is aware of the importance of bagasse waste from the sugar production industry which can be developed and used in research studies. To synthesize bagasse waste into aerogel absorbent material, the bagasse must be chemically treated with alkali to remove hemicellulose and lignin. In addition, the physical properties of aerogels can be adjusted by various synthetic methods for application in a variety of applications. The aerogel absorbent material is a low-density and highly porous material. In addition, the synthesis of aerogels has received much attention and is the ultimate value addition. The aerogel synthesis process involves several forming methods, including supercritical drying, freeze drying, and ambient pressure drying, etc. Supercritical drying is used for synthesis at high temperature and pressure conditions. This makes the method energy-intensive, dangerous, and expensive [2], thus limiting it. Freeze-drying uses a freezing technique in which the wet gel liquid is frozen and then dried by sublimation. But it takes a long time, maybe more than 2 days [3]. If this method is used, complex freeze-drying equipment is required. The above two processes are not suitable alternatives for industrial-scale aerogel production. However, using atmospheric pressure drying is a simple technique. Energy-saving and safe, cost-effective, and environmentally friendly, which does not require complicated equipment [4]. The researcher is aware of the importance of industrial waste that can be developed and used in research studies. Therefore, the idea is to find a way to synthesize cellulose waste from this industry into aerogels, which are absorbent materials, and develop them into pesticide-absorbent materials modified with zinc sulfide centers. The mixing of zerovalent zinc to increase the efficiency of pesticide treatment. Therefore, pesticides gain electrons through redox reactions. This results in changes in the structure of pesticides. This reduces the toxicity of pesticides [5] and will result in zinc rust. However, the rust problem can be solved by adding sulfide to the zinc synthesis process [6]. The study steps are as follows: Cellulose waste pretreatment, synthesis of aerogels from cellulosic waste using an ambient pressure drying process, synthesis of sulfide-modified zerovalent zinc aerogels, and characterization using various techniques including FTIR, SEM, XTM, and XRD.

Experimental/methodology.

Material and chemicals. Cellulose was used from waste products from the sugar production industry. Bagasse from Nakhon Ratchasima Province. Sodium hydroxide solution 99 percent (RCI Labscan), Hydrogen peroxide (purity 30 percent, Quality Reagent Chemical), Naphthalene (Himedia), Ethanol (Duksan), Acetone (RCI Labscan), Polyvinyl alcohol (Mw 85,000-124,000, 99+% hydrolyzed, SIGMA ALDRICH), Zinc Chloride (Kemaus), Sodium borohydride 97% (Loba chemie) and Sodium hydrosulfite (Alfa Aesar).

Instruments. We used four techniques to characterize the aerogel and sulfide-modified zinc aerogel: Scanning Electron Microscope (SEM- JEOL/JSM-6010LV), X-ray Diffractometer (Bruker, D2 Phaser), Fourier Transform Infrared Microscope Spectrophotometry (FT-IR Microscope/TENSOR 27) and Synchrotron X-ray tomographic microscopy at beamline 1.2W was operated at 1.2GeV, 150 mA in Synchrotron Light Research Institute (SLRI).

Pretreatment of bagasse. Raw bagasse is pretreated with sodium hydroxide at a temperature of 80˚C for 2 hours, then washed with deionized water until the pH is neutral. After that conditioned with hydrogen peroxide at a temperature of 80°C for 1 hour, then treated with sulfuric acid for 45 minutes, then washed with deionized water until the pH was neutral and dried at 80°C for 24 hours [7].

Synthesis of aerogels from cellulose waste. Using ambient pressure drying, prepare naphthalene ($C_{10}H_8$) and mix it with ethanol (C_2H_5OH) and acetone (C_3H_6O) stirring for 60 minutes, when the time is up, add the conditioned cellulose and polyvinyl alcohol solution, and stir for 30 minutes, and left at room temperature for 120 minutes. Finally, then bake at 70°C for 120 minutes and continue baking at 120°C for 120 minutes to obtain aerogels synthesized from cellulose waste [8].

Synthesis of sulfide-modified zero-valent zinc aerogel. The aerogels synthesized from sugarcane bagasse at ambient pressure were soaked in $ZnCl_2$ (0.1mol/L) for 24 hours, specially treated with 0.945 g of $NaBH_4$ and 0.043 g of $Na_2S_2O_4$, 50 ml of DI water, and then dropped into $ZnCl_2$ with aerogel at the rate of 1 drop was stirred for 30 minutes and purged with nitrogen gas. After that, wash with ethanol three times again and bake for 2 hours at 60°C [9]

Results and discussion.

From the research study, the synthesis of aerogel mixed with sulfide-modified zero-valent zinc, which consists of the bagasse pretreatment process, the process of synthesis of aerogels from cellulose waste, and the process of synthesis of sulfide-modified zero-valent zinc aerogel, shown as shown in **Figure 1**. However, an important part of this research is the study of properties, divided into two parts: first is the study of bagasse pretreatment characteristics. The properties were studied with various techniques: FTIR, SEM, and XRD. The second is the characterization of sulfide-modified zero-valent zinc aerogel which were studied with SEM and XTM.

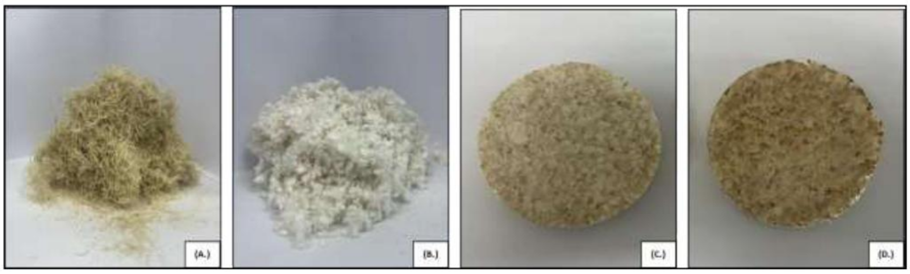

Figure 1. The synthesis process of aerogel mixed with sulfide-modified zero-valent zinc (A.) Raw bagasse (B.) Bagasse after pretreatment (C.) Aerogels from cellulose waste (D.) Aerogel mixed with sulfide-modified zero-valent zinc.

Bagasse pretreatment characteristics.

The properties were studied using various techniques including FTIR, SEM, and XRD. The results of the Fourier transform infrared spectrophotometer (FTIR) are shown in **Figure 2.** A peak at 2800-2900 cm^{-1} was found in both raw and pretreated bagasse samples, which were found to be C-H bonds of cellulose. A peak at 1598 cm^{-1} was found in only one raw sugarcane bagasse sample, which is a C–C bond, indicating the nature of the aromatic ring of lignin. A peak at 1,310 cm^{-1} was found in both raw and pretreated bagasse samples, indicating the characteristics of cellulose, and a peak at 1,161 cm^{-1} was found in only one raw sugarcane bagasse sample, which is a C=O bond, indicating the nature of hemicellulose [10] The results of the study found that when raw sugarcane bagasse is pretreated with a chemical process, it can reduce the amount of hemicellulose and lignin in raw sugarcane bagasse.

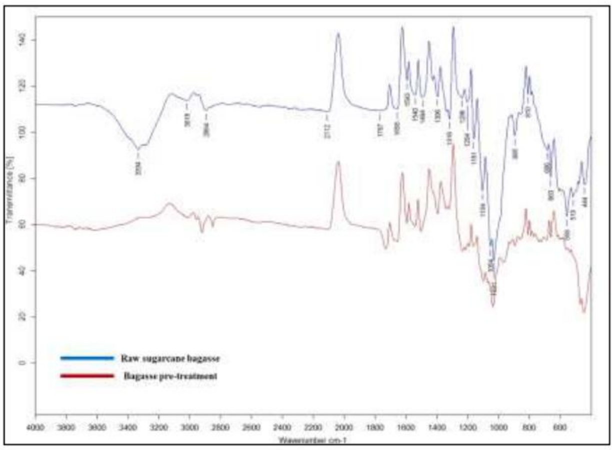

Figure 2. Results of Fourier-transform infrared spectroscopy technique of bagasse waste pretreatment

The results of the scanning electron microscope (SEM) are shown in **Figure 3.** Scanning electron microscopy was used to study the surface of the samples to study the morphology of two samples, both raw and pretreated bagasse. Chemical treatment with sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), and sulfuric acid (H_2SO_4), where pretreatment can help remove impurities from raw sugar cane fibers on the surface.

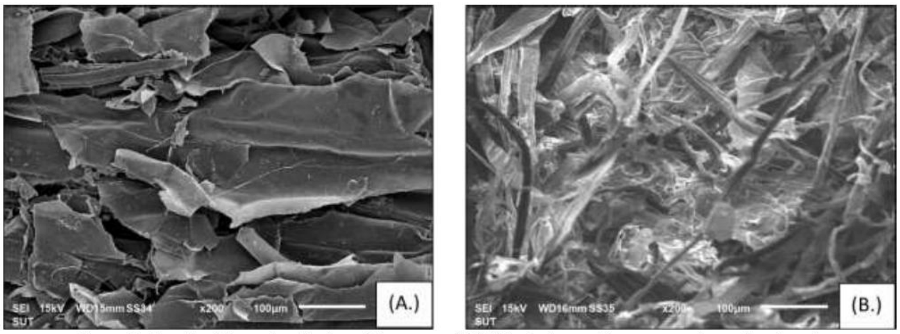

Figure 3. Results of Scanning Electron Microscope (SEM) technique of bagasse waste pretreatment
(A.) Raw bagasse (B.) Bagasse waste pretreatment

The results of the X-ray Diffractometer (XRD) study are shown in **Figure 4.** To study the crystal structure of two samples, raw sugarcane bagasse and chemically treated bagasse. This is because cellulose has a crystalline structure, while hemicellulose and lignin are amorphous. The crystalline peak has a 2θ value of 220 or the 002 plane, and the amorphous peak has a 2θ value of 160 or the 101 plane. From the results of the research, it was found that after pretreatment, the crystallinity value was higher than that of untreated raw sugarcane bagasse. This is consistent with the results of molecular structure analysis using the FTIR technique, which confirmed that lignin and hemicellulose can be reduced [11]. The XRD results can be converted to crystallinity index values (Crystallinity Index; CrI) calculated in Equation (1)

$$\text{Crystallinity index (\%)} = [\,(I_{002} - I_{101}) / I_{002}\,] * 100 \quad (1)$$

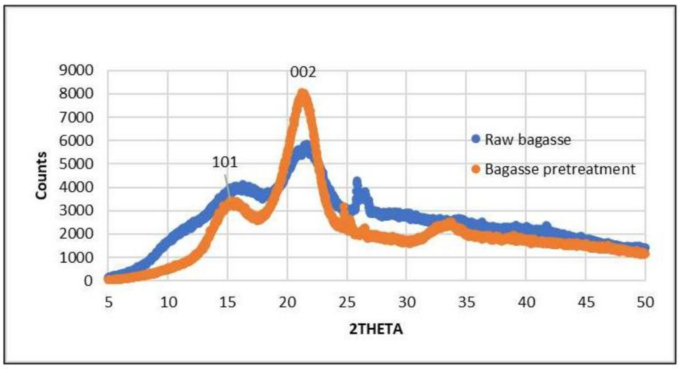

Figure 4. Results of X-ray Diffractometer (XRD) technique of bagasse waste pretreatment (A.) Raw sugarcane bagasse (B.) bagasse waste pretreatment

Table 1 The X-ray Diffractometer (XRD) results show the crystallinity index. (Crystallinity index; CrI) of raw and pretreated bagasse.

Sample	Crystallinity index; CrI
(A.) Raw bagasse	42.95
(B.) Pretreated bagasse waste	67.41

From **Table** 1 , calculate the crystallinity index according to **Equation 1**. It was found that the crystallinity index value when comparing raw bagasse and bagasse treated with NaOH, H_2O_2, and H_2SO_4 had a higher crystallinity index value indicating an increased proportion of cellulose after pretreatment. This shows that with chemical pretreatment the amorphous part can be removed [12].

Characteristics of sulfide-modified zero-valent zinc aerogel.

The properties were studied using SEM and XTM techniques. The surface morphologies of sulfide-modified zero-valent zinc aerogel by the scanning electron microscopy (SEM) technique are shown in **Figure 5.** The SEM results in **Figure** 5 **A**, there were visible gaps between the bagasse fibers, but in **Figure 5B**, the fibers were not visible because the aerogel was coated with sulfide-modified zero-valent zinc.

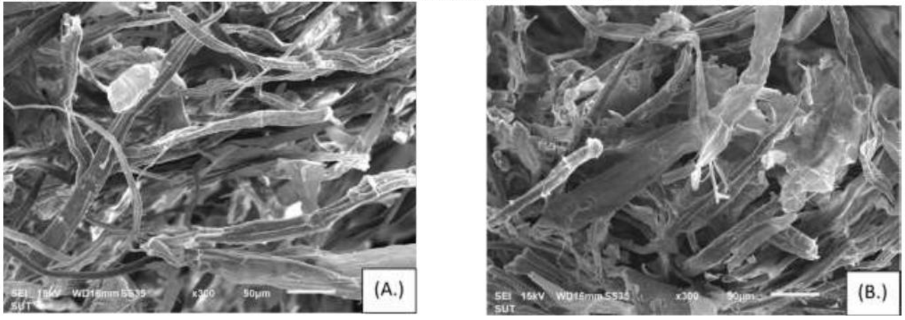

Figure 5. Results of scanning electron microscopy (SEM) technique of the synthesis process of sulfide-modified zerovalent zinc aerogel (A.) Aerogel (B.) Sulfide-modified zerovalent zinc aerogel

X-ray 3D characteristics and porosity (%) of sulfide-modified zerovalent zinc aerogels were studied using the X-ray Tomographic Microscopy (XTM) technique, the results are shown in **Figure 6.** In this research, aerogel samples synthesized from bagasse cellulose and aerogel mixed with sulfide-modified zero-valent zinc were studied. The XTM results in **Figure 6A**, show a 3D tomographic and porosity of 88%, and in **Figure 6B,** the porosity of 70% because the aerogel was coated with sulfide-modified zinc ionic centers. As a result, the porosity value decreases.

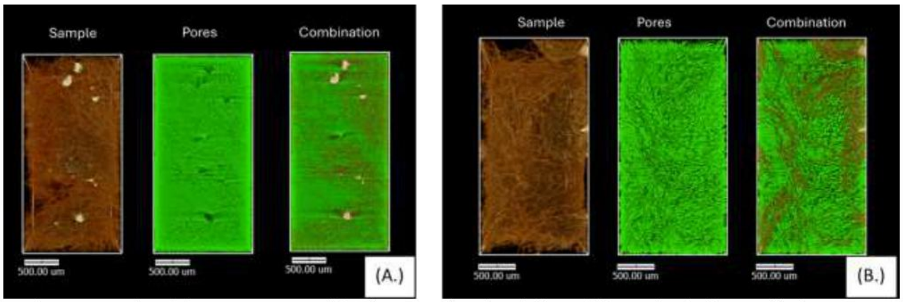

Figure 6. Results of the X-ray Tomographic Microscopy (XTM) technique of the synthesized aerogel (A.) Bagasse aerogel (B.) Sulfide-modified zerovalent zinc aerogel.

Conclusions.

This research studies the synthesis of aerogels from sugarcane bagasse waste, which is chemically adjusted with sodium hydroxide, hydrogen peroxide, and sulfuric acid to remove lignin, and hemicellulose contaminants from bagasse fibers. Then its properties were investigated using various techniques, namely FTIR, SEM, and XRD. The results of (FT-IR) found that the chemically pretreated bagasse sample was able to remove lignin and hemicellulose, which was consistent with the XRD result. It was found that the crystallinity index of cellulose increased for the chemically pretreated bagasse sample. From SEM images, it is found that when chemically adjusted it was able to remove impurities from the fibers. After that, the bagasse cellulose was synthesized into an aerogel absorbent material mixed with sulfide-modified zero-valent zinc through an ambient pressure drying process. Later, the properties were studied using SEM and XTM techniques. The SEM technique found that the surface area characteristics of the aerogel were those of the mixed zinc modified with sulfide. The fibers are not visible because the aerogel is coated with sulfide-modified zerovalent zinc. The results of the XTM technique revealed that when the aerogel was coated with sulfide-modified zinc, the porosity decreased from 88% to 70%.

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