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Abstract	The present research work focused on the extraction of natural dye from Mangosteen peel (Garciniamangostana Linn). The physico-chemical properties such as moisture content, ash content, total solids content and pH of Mangosteen peel were determined by association of official Analytical Chemists (AOAC) method. The chemical compounds present in Mangosteen peel were investigated by phytochemical tests. Natural dye was extracted from Mangosteeen peel with pure water at 70ºC for 60 minutes. The physico-chemical properties of extracted natural dye were also determined by AOAC method. An attempt was made on the dyeing of cotton fabrics using extracted natural dye with three different mordants such as zinc sulphate, potassium dichromate, potash alum and three different dyeing methods such as pre-mordanting, post-mordanting and simultaneous mordanting and different dyeing methods on the properties of dyed cotton fabrics were also investigated. Moreover, washing and rubbing fastness properties, staining and colour change of dyed cotton fabrics were assessed by using standard Grey scale.
Keywords	Groundnut shell, activated carbon, fish sauce, decolourization
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An Investigation on the Effectiveness of Prepared Activated Carbon from Lignocellulosic Waste (Groundnut Shell) on the Decolourization of Fish Sauce

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Abstract

Environmental friendly activated carbon from lignocellulosic waste, groundnut shell was processed by chemical activating method. Before carbonization, the precursor was impregnated in 20% calcium chloride solution. The highest fixed carbon content, 78.5% was obtained for processed groundnut shell activated carbon. Carbonization temperature at 500°C and carbonization period for 60 min gave the highest fixed carbon content.Yield percentage of the activated carbon was 68%. The nature of processed and commercial activated carbons was analyzed by X-ray Diffraction (XRD) method. The surface area, 349.6m^2/g was obtained with the iodine sorption capacity 76% and methylene blue number 170mg/g.

By using processed activated carbons as well as commercial activated carbon, fish sauce from Bago Township, Bago Region was decolourized. Percentages of bleaching efficiency of activated carbons were determined. Characteristics of decolourized fish sauce such as pH, specific gravity, colour, soluble solids content, sodium chloride content, protein content and total plate count were investigated. The highest bleaching efficiency was formed with 1: 40 (weight ratio) groundnut shell activated carbon to fish sauce at room temperature for 3hr. contact period and it was observed that characteristics of decolourized fish sauce were same as market product, Thai made fish sauce.

Keywords: groundnut shell, activated carbon, fish sauce, decolourization

Introduction

Activated carbon (AC) is a non-graphitiable carbon with a highly disordered microstructure. It is well known for high adsorption capacity due to its high surface area and porosity. An activation level sufficient for useful application may be attained solely from high surface area; however, further chemical treatment often enhances adsorption properties. Activated carbon is usually derived from charcoal and is sometimes utilized as biochar. Those derived from coal and coke are referred as activated coal and activated coke respectively (en wikipedia.org/wiki/Activated Carbon).

Among various adsorbents used, activated carbon is well known for its high adsorption capacity due to large surface area and pore volume. In recent years, immerse research has been focused towards converting the agricultural or lignocellulosic wastes into activated carbon, since this technology not only solve the problem of waste disposal but also convert a potential waste into a valuable product that can be used as an adsorbent for effluent treatment. Activated carbon can be produced from any carbonaceous materials, both naturally occurring and synthetic (John, W.H., 1974).

Carbonization is a process in which the carbonaceous materials are decomposed by heat so as to turn the organic substances into carbon and remove any non-carbon typed materials. It is a process to remove those hydrocarbons which may block the pores of charcoal. Activated carbon (AC) is the carbonaceous material which plays an important

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role in adsorption process. Its ability is to remove organic and inorganic wastes, odour, colour and taste from any kind of chemical industry process is based on their amazing properties. Besides, it has higher surface area and micro porous structure. Prior to carbonization, the raw material is impregnated with certain chemicals (strong base,acid or a salt). Chemical activation is preferred over physical activation owing to the lower temperatures and shorter times needed for activating materials (Ketcha, J. M. *et al*., 2012).

The advantage of activated carbon materials as adsorbents is that the treated effluent is of high quality, the design of the process is simple, the operation of the process developed or adopted is easy. In addition to purification of gases and liquids with high adsorption potential, activated carbon materials are also used as catalysts and catalyst supports (Viswanathan, B., *et al*., 2009).

Today, activated carbon is used in a wide range of industrial applications. Activated carbon has also been used to an increasing extent to produce decolourized fish sauce. In Myanmar, Ngan Pya Ye (Fermented Fish Sacue) is a dark brown colour extractor sauce obtained as a byproduct of Nga-pi (fermented fish) manufacture. Heating with activated carbon together with continuous stirring is used for decolourization of fish sauce. Using this method, the colour and part of the odour can be removed simultaneously. Ngan Pya Ye (fermented fish sauce) is part of the traditional diet of Myanmar people. Their nutritive value as a protein to supplement the protein-poor diet of the Myanmar is of great importance. Myanmar fish sauce has high content of sodium chloride and its colour and smell is also unattractive to the consumers.

Thus the objective of this study is to prepare environmental friendly adsorbents, from lignocellulosic waste (groundnut shell) and use in decolourization of local fish sauce.

Materials and Methodology

Materials

Groundnut shells were collected from Laywagyi Village, Myinmu Township, Sagaing Region. Commercial activated carbon and calcium chloride (Japan made, Analytical grade,) were purchased from Golden Lady Chemical Sale Center located at (28^{th}) Street, Pabedan Township, Yangon Region. Fish sauce from Bago Township, Bago Region was decolourized with activated carbons.

Methods

Preparation of Raw Materials

Groundnut shells were washed with tap water to remove earthy matter and sun dried for 24 hr. The dried shells were cut into small pieces (1.5cm x 0.5 cm).

Physico-chemical Characteristics of Raw Materials

Physico-chemical characteristics such as moisture content, volatile matter content, ash content, bulk density and fixed carbon content of groundnut shells were determined. (ASTM –D1762)

Preparation of Activated Carbon

Chemical Activation of Dried Raw Materials

5 g of cleaned and dried groundnut shells were impregnated in 10 mL. of 20% $CaCl_2$ solution and then soaked for overnight at room temperature. Then the samples were sieved with a stainless steel sieve and put into the crucible and dried in a hot air oven at 105°C for 30 min before carbonizing in a muffle furnace.

Carbonization of Chemically Activated Raw Materials

5 g of chemically activated raw materials were carbonized in a muffle furnace (Lab Tech, LEF-1155-2) at various temperatures for 120 min carbonization period. The most suitable carbonized materials were selected and the most preferable carbonization temperature was chosen. To get the most suitable and economically reliable carbonization

period, raw materials were again carbonized at chosen temperature for 30 min, 45 min, 60 min, 75 min, 90 min, 105 min and 120 min.

Washing of Activated Carbon

After carbonization, activated carbon was cooled down and taken out carefully from muffle furnace. Then, it was washed with water for six times to remove trace chemicals. After washing, it was kept on the tray, and transferred into the oven at 110°C for 2 hr. and then put into airtight bag and stored in desiccators.

Determination of Yield Percentage of Activated Carbon

The yield of activated carbon was calculated on a chemical-free basis and can be regarded as an indicator of the process efficiency for the process. The yield of activated carbon was calculated as the percentage weight of the resultant activated carbon divided by weight of dried precursor used.

Characterization of Activated Carbon

Physico-chemical Characteristics of Activated Carbon

Physico-chemical characteristics of prepared and commercial activated carbons such as moisture content, volatile matter content, ash content, and fixed carbon content were determined. (AWWA- 1991, Standards for Granular Activated Carbons)

X-ray Diffraction Spectroscopy (XRD) Analysis

X-ray Diffraction Spectroscopy, an analysis of processed activated carbon was carried out with Powder X-ray Diffractometer, Multiflex Rigaku, Graphite momochromator. Similarly, the nature of commercial activated carbon was characterized to compare the processed activated carbons by XRD analysis. This analysis was conducted at the Laboratory of Universities Research Centre, University of Yangon.

Determination of Surface Area, Porosity and pH of Activated Carbon

Surface area, iodine sorption capacity (Okibe, F.G., *etal*, 2013), methylene blue number (Ceiton, A.N., *etal*, 2011) and pH of activated carbon was determined. pH of activated carbon was measured by using pH meter (HANNA pH 300, Germany Made).

Characterization of Raw Fish Sauce

Physico-chemical properties like pH, specific gravity, and soluble solids content were determined by AOAC Methods. Sodium chloride and protein content of fish sauce were also determined (Pearson, 1981). The colour of fish sauce was determined by Spectrophotometer (TRSP-722). Furthermore, bacteriological examination as total plate count of raw fish sauce was also detected.

Decolourization of Fish Sauce by Activated Carbon

Various ratios of activated carbon to fish sauce were treated by using processed activated carbon and commercial activated carbon at room temperature for 2hr with 500 rpm stirring rate. The most suitable ratio of activated carbon to fish sauce was selected and the most preferable contact time and temperature were further examined.

Characterization of Decolourized Fish Sauce and Commercial Fish Sauce

Characteristics of decolourized fish sauce and commercial fish sauce were determined as the same procedures used to determine the characteristics of raw fish sauce.

Results and Discussion

Characteristics of raw materials for preparation of activated carbon are described in Table (1). As the lignocellulosic waste is carbonaceous material, fixed carbon content of groundnut shell was found to be that of 54.4 %. It was observed that moisture content was 28.68 w/w% but ash content of groundnut shell was 1.21 w/w%. The high fixed carbon content and low ash content indicate that this material was suitable source for preparation of activated carbon. Volatile matter content was 15.71% with 0.11 kg/m^3 bulk density. The characteristics of activated carbon mainly depend upon the physical and chemical properties of raw materials.

Effect of carbonization temperature on the physico-chemical properties of activated carbon prepared from groundnut shell by using impregnation agent, $CaCl_2$ are described in Table (2). Fixed carbon content increased with increase of carbonization temperature up to certain value and then it was decreased. The highest fixed carbon content 60.48% was obtained for groundnut shell activated carbon at 500°C for carbonization period 60 min. It is the most important parameter for determination of quality of activated carbon. With the increase of carbonization temperatures from 200°C to 600°C, the yield of prepared AC - $CaCl_2$ decreased from 60% to 26%.

Effect of carbonization time on the properties of prepared activated carbons is shown in Table (3). The carbonization period at 500°C was varied in between 30 min to 105 min (30, 45, 60, 75, 90, and 105 min) to investigate its effect on physico-chemical properties. Fixed carbon content increased with increase of carbonization period up to certain value and then gradually decreased. Whereas ash content decreased with increase of carbonization time up to certain value and then increased. The highest fixed carbon content 78.5% was observed with 7.3 % ash content. Thus the most the most suitable carbonization period was 60 min at the most the most suitable carbonization temperature, 500°C. The yield of activated carbon was decreased at longer carbonization period because of the higher fixed carbon content and changed to form as char. It could be attributed to the release of more volatiles by keeping the sample precursor for larger duration at final carbonization temperature.

Physico–chemical properties of prepared activated carbon and commercial activated carbon are indicated in Table (4). Moisture content of prepared activated carbon was 6.1%. For many purposes, this moisture content does not affect the adsorptive power, but obviously it dilutes the carbon. Volatile matter is usually specified free of moisture content and for processed activated carbon 8.1% but 4.85% for commercial activated carbon. The inorganic material contained in activated carbon is determined as ash content. Bulk density is used to determine the weight of a fixed volume of activated carbon. The bulk density of activated carbon depends on the shape, size and density of the individual particles. Bulk density of processed activated carbon was 0.17 kg/m^3 but commercial activated carbon was 0.30 kg/m^3.

The fixed carbon content was 78.5% for processed activated carbon and 65.19% for commercial activated carbon. The characteristics of activated carbon thus depend on the physico-chemical properties of raw material, carbonization temperature and time as well as chemical activation agent used. Due to chemical activation before carbonization, activated carbon can be processed at lower temperature with shorter carbonization time. The nature of prepared activated carbon and commercial activated carbon were analyzed by X-ray diffraction method and results are described in Fig. (1). X-ray diffraction analysis demonstrated that both processed activated carbons and commercial activated carbon possessed graphitic character and low content of inorganic constituents.

Iodine sorption capacity presented micro pore and meso pore content of activated carbon. The surface area and micro and meso pore content are related because these two determination procedures indicated that processed activated carbon which had more micro and meso pores, more surface area they had. Surface area and porosity of activated carbons are shown in Table (5).

Local fish sauce (Ngan Pya Ye), which usually has a dark brown colour, is not attractive for table use. Although it has an export potential, its colour and strong smell are usually offensive to foreign customers. The brown colour of fish sauce processed by traditional method is caused by non-enzymatic browning reaction. Characteristics of raw fish sauce from Bago Township are described in Table (6). It contained 3.51% protein content with high sodium chloride content 43.45%. More than10^4cfu/mL of total plate count were found in raw fish sauce.

Decolourization of fish sauce by different ratios of activated carbon to fish sauce is described in Table (7). It was observed that the most suitable condition of bleaching efficiency occurred at ratios of 1:40 groundnut shell activated carbon to fish sauce, and 1:30 commercial activated carbon to fish sauce. By using these ratios, the sodium chloride contents were found to be with minimum. Thus, processed activated carbon exhibited superior colour adsorption as compared to commercial activated carbon. The most suitable ratios of activated carbon to fish sauce were again used for decolourization at longer contact periods and data are shown in Table (8). The highest colour removal percent, 23% with least sodium chloride content, 30% were observed at 3hr. contact period, for 1:40 processed activated carbon to fish sauce, and 1:30 commercial activated carbon to fish sauce respectively. The most suitable ratios of activated carbon to fish sauce with the most suitable contact time were further treated at higher temperature and results are shown in Table (9). It was clearly observed that the bleaching efficiency gradually decreased as the temperature was increased. The colour of the fish sauce became dark and the product qualities were deteriorated at higher temperature.. But sodium chloride content decreased with the increasing temperature. Thus the most suitable contact temperature to treat fish sauce by activated carbon was room temperature.

The quality of decolourized fish sauce was compared to commercial fish sauce as shown in Table (10). In this research, standardized fish sauce was obtained by bleaching and partial deodourization with processed activated carbon as well as with commercial activated carbon. But lesser amount of processed activated carbon was required to get a standardized fish sauce than commercial activated carbon. The colour of the decolourized fish sauce changed from deep brown to golden brown and pungent odour and sodium chloride content were simultaneously reduced. The colour of the decolourized fish sauce became lighter and more transparent as the market product, Thai made fish sauce. But the sodium chloride contents in decourized fish sauce were slightly higher than Thai made fish sauce and this may be originally high salt content of traditionally processed fish sauce. Protein contents of decolourized fish sauce were also higher than market product and this may be because of the composition of raw ingredients of fish sauce. The total plate count of fish sauce was reduced from more than 10^4cfu/mL to 5 x 10^3cfu/ after decolourization.

Table (1) Characteristics of Groundnut Shell for Preparation of Activated Carbon

Sr. No	Characteristics	Experimental Value
1	Moisture (w/w %)	28.68
2	Volatile matter (w/w %)	15.71
3	Ash (w/w %)	1.21
4	Bulk density (kg/m^3)	0.11
5	Fixed carbon content (w/w %)	54.4

The experiments were conducted at the Laboratory of Department of Industrial Chemistry, Yadanabon University.

Table (2) Effect of Carbonization Temperature on the Physico-chemical Properties of Activated Carbon from Groundnut Shell

Activating agent = $CaCl_2$
Carbonization period = 60 min.
Weight of raw materials = 5 g
Impregnation ratio of raw material and activating agent = 1: 2

Sr. No.	Carbonization temperature (°C)	Physico-chemical Properties				Yield percentage of activated carbon (w/w %)
		Moisture content (w/w %)	Volatile matter content (w/w %)	Ash content (w/w %)	Fixed carbon content (w/w %)	
1	200	12.21	9.28	12.42	64.11	60
2	300	33.71	10.01	14.50	41.78	31
3	400	34.46	8.56	14.18	42.80	38
4	500*	11.03	12.71	15.78	60.48	35
5	600	11.94	7.31	21.66	59.03	26

* = most suitable carbonization temperature

Table (3) Effect of Carbonization Period on the Physico-chemical Properties of Activated Carbon from Groundnut Shell

Activating agent = $CaCl_2$
Carbonization temperature = 500°C
Weight of raw material = 5 g
Impregnation ratio of raw material and activating agent = 1: 2

Sr. No.	Carbonization period (min)	Physico-chemical Properties				Yield percentage of activated carbon (w/w%)
		Moisture content (w/w%)	Volatile matter content (w/w%)	Ash content (w/w%)	Fixed carbon content (w/w%)	
1	30	7.6	7.3	7.9	77.2	84
2	45	7.5	7.2	8.0	77.3	78
3	60*	6.1	8.1	7.3	78.5	68
4	75	6.1	9.1	8.1	76.7	66
5	90	8.1	10.0	7.1	74.8	56
6	105	9.1	12.0	8.1	70.8	54

* = most suitable carbonization period

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Table (4) Physico–chemical Properties of Prepared Activated Carbon and Commercial Activated Carbon

Carbonization temperature = 500 °C

Carbonization period = 60 min

Sr.No.	Properties	Activated carbon from groundnut shell	Commercial activated carbon
1	Moisture (w/w %)	6.1	8.05
2	Volatile matter (w/w %)	8.1	4.85
3	Ash (w/w %)	7.3	20.01
4	Bulk density (kg/m^3)	0.17	0.30
5	Fixed carbon content (w/w %)	78.5	65.19
6	pH	6.29	6.82

Table (5) Surface Area and Porosity of Prepared Activated Carbons

Sr. No	Parameter	Activated carbon from groundnut shell	Commercial activated carbon	Literature value*
1	Surface area (m^2/g)	349.6	259.3	199-2105
2	Iodine sorption capacity (w/w%)	76.0	72.0	76-84
3	Methylene blue number (mg/g)	170	167	0-501

* = (Okibe, F.G., *et al.,* 2013)

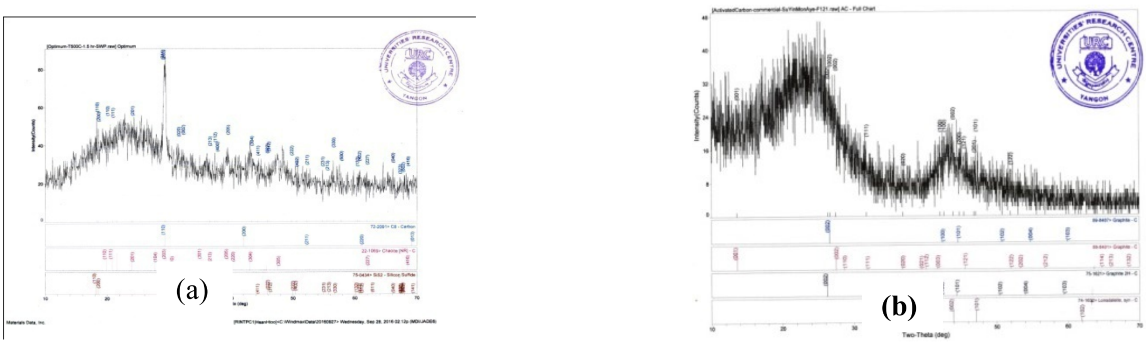

Figure (1) X-ray Diffractogram of (a) Groundnut Shell Activated Carbon (b) Commercial Activated Carbon

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Table (6) Characteristics of Raw Fish Sauce from Bago Township

Sr. No.	Characteristics	Experimental Value	Literature Value
1	pH	5.61	5-6.5
2	Specific gravity	1.23	1.2
3	Colour (absorbance)	1.801	-
4	Soluble solids content (ºBrix)	31.00	-
5	Sodium chloride content (w/w %)	43.45	24
6	Protein content (w/w %)	3.51	10
7	Total plate count (cfu/mL)	greater than 10^4	-

Literature value is based on Codex Standard for Fish Sauce (CODEX STAN 302-2011)

Table (7) Effect of Ratio of Activated Carbon to Fish Sauce on Bleaching Efficiency and Sodium Chloride Content

Temperature = room temperature
Contact period = 2 hr
Stirring rate = 500 rpm
Colour of raw fish sauce (absorbance) = 1.801
Sodium chloride content of raw fish sauce (w/w%) = 43.45

Sr. No.	Activated Carbon : Fish Sauce	Decolourized Fish Sauce					
		Groundnut Shell Activated Carbon			Commercial Activated Carbon		
		Colour (absorbance)	BE (%)	NaCl (w/w %)	Colour (absorbance)	BE (%)	NaCl (w/w
1	1:150	1.60	11.16	41.20	1.73	3.94	42.20
2	1:75	1.590	11.72	40.25	1.6	11.16	40.20
3	1:50	1.582	12.16	39.05	1.58	12.27	39.2
4	1:40	1.47*	18.38*	38.5*	1.50	16.71	39.0
5	1:30	1.47	18.38	38.45	1.48*	17.82*	38.5*
6	1:25	1.472	18.39	38.0	1.48	17.82	38.5

*Suitable condition, BE- Bleaching Efficiency

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Table (8) Effect of Contact Period on Bleaching Efficiency and Sodium Chloride Content of Activated Carbons

Ratio of groundnut shell activated carbon to fish sauce = 1: 40
Ratio of commercial activated carbon to fish sauce = 1: 30
Temperature = room temp:
Colour of raw fish sauce (absorbance) = 1.12
Sodium chloride content of raw fish sauce (w/w%) = 40.45

Sr. No.	Time (hr)	Decolourized Fish Sauce					
		Groundnut Shell Activated Carbon			Commercial Activated Carbon		
		Colour (absorbance)	BE (%)	NaCl (w/w %)	Colour (absorbance)	BE (%)	NaCl (w/w%)
1	1	1.02	8.92	38.48	1.00	10.71	38.78
2	2	0.94	16.07	35.20	0.95	15.18	33.86
3	3*	0.86	23.21	30.18	0.86	23.21	30.22
4	4	0.87	22.32	30.11	0.86	23.21	30.34
5	5	0.87	22.32	30.15	0.87	22.32	30.17
6	6	0.86	23.21	30.12	0.87	22.32	30.15

*Suitable condition, BE- Bleaching Efficiency

Table (9) Effect of Temperature on Bleaching Efficiency and Sodium Chloride Content of Activated Carbons

Ratio of groundnut shell activated carbon to fish sauce = 1: 40
Ratio of commercial activated carbon to fish sauce = 1: 30
Contact period = 3 hr
Colour of raw fish sauce (absorbance) = 1.12
Sodium chloride content of raw fish sauce (w/w%) = 42.45

Sr. No.	Temp; (ºC)	Decolourized Fish Sauce					
		Groundnut Shell Activated Carbon			Commercial Activated Carbon		
		Colour (absorbance)	BE (%)	NaCl (w/w %)	Colour (absorbance)	BE (%)	NaCl (w/w%)
1	RT*	0.86	23.21	30.18	0.86	23.21	30.72
2	35	0.89	20.54	29.15	0.87	22.32	25.32
3	40	0.9	19.64	29.00	0.89	20.54	22.68
4	45	0.93	16.96	28.88	0.92	17.85	22.42
5	50	0.95	15.78	27.65	0.96	14.29	21.56

RT= room temperature, *Suitable condition, BE- Bleaching Efficiency

Table (10) Comparison of the Properties between Decolourized Fish Sauce and Commercial Fish Sauce

Sr. No.	Properties	Decolourized Fish Sauce		Fish Sauce (Thai Made) Squid Brand	Literature Value
		Groundnut Shell Activated Carbon	Commercial Activated Carbon		
1	pH	5.62	5.59	5.72	5-6.5
2	Specific gravity	1.21	1.21	1.19	1.2
3	Colour (absorbance)	0.86	0.86	0.82	-
4	Soluble solids content (ºBrix)	35.40	35.40	33.28	-
5	Sodium chloride content (w/w%)	30.18	30.72	24.22	24
6	Protein content (w/w %)	6.51	6.49	5.80	10
7	Total plate count (cfu/mL)	9 x10^3	4 x10^3	2 x10^3	-

Literature value is based on Codex Standard for Fish Sauce (CODEX STAN 302-2011)

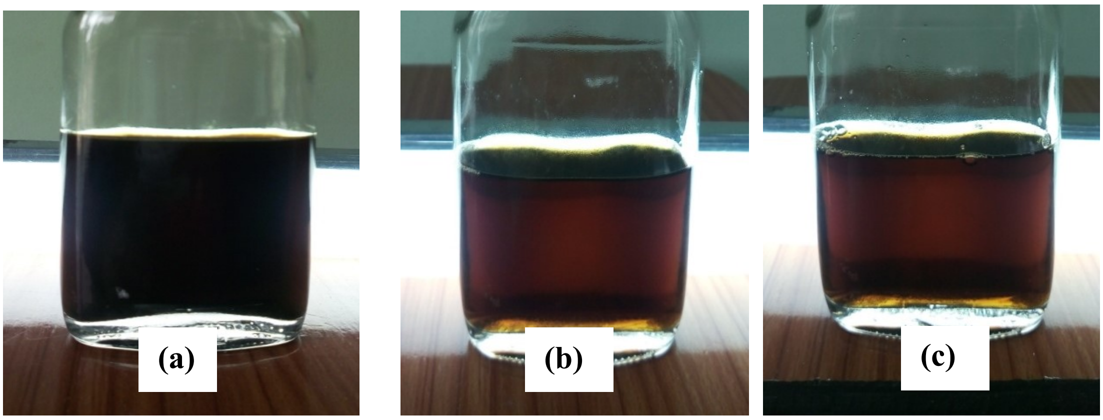

Fig (2) (a) Raw Fish Sauce, (b), (c) = Decolourized Fish Sauce using; Groundnut Shell Activated Carbon, Commercial Activated Carbon

Conclusion

In addition the choice of a cheap precursor for the production of activated carbon means both considerable saving in production cost and a way of making use of waste materials, thus reducing its disposal and environmental polluted problem. Owing to chemical activation, precursors could be activated at lower temperatures and shorter time. Activation does help to enlarge the process of the carbonized materials, hence increased in adsorption power. Fixed carbon content plays an important role where the carbon surface holds the adsorbate molecule. This higher fixed carbon content activated carbon, 78.5% from groundnut shell had large surface for adsorbate to be adsorbed than commercial activated carbon, 65.19% fixed carbon content. The experimental results indicated that this method of preparation of activated carbon was easy, economic and prepared activated carbons have very significant physico-chemical properties.

Decolourization with groundnut shell activated carbon was the most economic one since it was needed in less of amount than the commercial at the same treatment temperature and time. The nutritional and flavour aspects of decolourized fish sauce were not adversely affected by decolourization. The colour of the decolourized fish sauce reduces from dark brown to golden brown and moreover, the pungent odour was significantly removed. Sodium chloride content of fish sauce was decreased simultaneously to a certain value. The characteristics of treated sauces were in agreement with Thai made (Squid brand) fish sauce.

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