Original Article

Verification of Sampling Media for Volatile Organic Compound (VOC) Using Local Source Activated Carbon from Agricultural Waste

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ABSTRACT : *Volatile Organic Compounds (VOC) are commonly monitored as they are a key contributor to photochemical smog. Many are hazardous to human health, with several classified as carcinogenic. The key sources of VOCs are industrial processes (especially those involving solvents) vehicle emissions, evaporative loss from petrol storage and even natural sources like forest fires. A group of VOCs, collectively known as BTEX, comprises benzene, toluene, ethylbenzene and xylene. To absorb the BTEX in the air, a sorbent tube using charcoal is one of the techniques. The objective of this study is to identify the agricultural waste from local sources that could be converted to activated carbon for VOCs sampling media and to verify the sampling media including detection limit, sensitivity, specificity, bias, accuracy, precision, recovery, maximum, volume, minimum volume and desorption efficiency. Hence, this study explored the methods of producing activated carbon from the precursor of palm shell, coconut shell, rubber seed shell and bamboo. For activated carbon development, the carbonization process occurs on the burner designated for cleaning the combustion of raw material in particular for large capacity up to 5 kg per production. Then the raw material is converted to charcoal by using a burner before it is sieving to a certain size for activation. The Laboratory at Universiti Tun Hussein Onn Malaysia (UTHM) and accredited laboratory has been used for activated carbon and sorbent tube testing. NIOSH Manual of Analytical Methods (NMAM), Hydrocarbon, Aromatic: Method 1501 has been used as main guidance for laboratory testing, verification and validation. The result shows that coconut shell activated carbon is the best VOCs sampling media followed by rubber seed shell, palm shell and bamboo. In conclusion, 4 local sources previously known as agricultural waste could be converted to raw materials for VOCs sampling media. The result of verification analysis and validation from the accredited laboratory show that the sampling media could be used and it similar to existing market sampling media such as the dimension of the sampling media which can used the existing tools for sample collection.*

Keywords: *VOC, Activated Carbon, Sorbent tube, BTEX, Monitoring. Safety and Health.*

1.0 INTRODUCTION

Demanding of fresh, healthy and comfort indoor environment is essential since most people spend their time indoor as compared to outdoor (Abechi *et al.,* 2013). The increasing concern towards indoor air quality (IAQ) has gained suitable techniques for mitigating the indoor air contaminant (Yu *et al.,* 2014) which can affect health and working performance (Yu *et al.,* 2009). Volatile Organic Compound (VOC), Carbon monoxide (CO), Carbon dioxide (CO_2), and particulate matter (PM_{10}) are defined as the major contributors to indoor air contaminants. There are several VOCs in indoor air environments such as BTEX (benzene, toluene, ethylbenzene and xylene), trichloroethylene and dichloromethane (Das *et al.,* 2004). In the long term, exposure may poses adverse health effects related to the human respiratory system (such as asthma, throat irritation, lung cancer), headache, poor memory, eyes, nose and skin irritation (Jo and Chun, 2014, Eugenija *et al.,* 2009). The use of sorbent tube to concentrate and store volatile compounds. The selection of which sorbent to use and best practices for managing high relative humidity are important considerations to allow for reproducible, untargeted, biomarker discovery in water saturated breath samples (Wilkinson et al., 2020). A large variety of different sorbent materials are available to researchers (Woolfenden 2010). Sorbent tubes can be purchased ready packed or made inhouse and require thorough thermal conditioning before use. A list of different sorbent materials that have been used to pre-concentrate and store VOCs. In recent years, the vehicle and indoor environmental standards are open to the selection of sorbent, no longer limiting the type of sorbent sorbent, and more sampling tubes mixed with different sorbent types have emerged to achieve the best sampling results (Hu et al., 2023). Microspore formation in AC formed during the burning process of AC. According to Bhadra et al., (2016) and Seo et al., (2016), Activated carbons (ACs) are well-known conventional adsorbents with many applications due to their hydrophobicity, surface functionality, pore structure, and high surface area. The raw materials that undergo burning process will be converted to charcoal. In this process the particle structure of the raw material will be restructure. Activated carbon (AC)was prepared from date palm leaflets using KOH activation followed by nitric acid oxidation to produce oxidized activated carbon (OAC) which possesses acidic and increase in pore number (Said et al., 2016). The bond between each particle will be break and change into new bond which will create the porous of the material. However, the present spore does not effective as the activated carbon since the number of microspores is less. Iovino et al., (2015) highlighted from study which mentioned that the sorbent is mainly microporous (with a micropore volume equal to 0.31 cm3 g-1). Figure 1.1 illustrate the pore formation on AC. The elemental force causing physical adsorption on activated carbon is the London dispersion force, a form of Van der Waals force, resulting from intermolecular attraction. In the case of adsorption, carbon and the adsorbate are thus chemically unchanged. However, in the process known as chemisorption, molecules chemically react with the carbon's surface (or an impregnant on the carbon's surface) and are held by chemical bonds that are much stronger forces compared to London dispersion forces. The London dispersion force is an intermolecular interaction that exists between all molecules (both polar and non-polar), but it is extremely short ranged. Jia et al., (2016) state that the rich AC has a characteristic that may be favorable for the adsorption of cationic species such as dyes through electrostatic interaction.

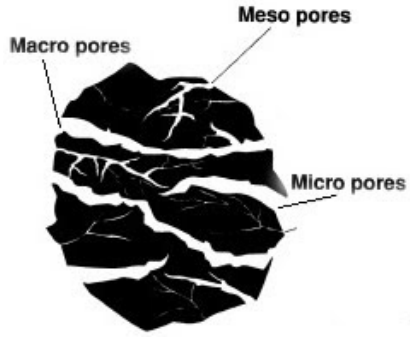


Figure 1.1: Pore Formation in AC

1.1 BTEX and its Effect on Human Health

Benzene, toluene, ethylbenzene and xylene (BTEX) are the most common VOCs found in various places and are classified as air pollutants that are hazardous to humans (Ertan et al., 2010, Nor Rahafza Abdul Manap *et al.,* 2018). It is volatile and easy to evaporate at a temperature higher than 20°C. The largest contributors of BTEX to the atmosphere were obtained from petrochemical fuel derivatives. Combustion of gasoline and diesel fuels especially for vehicles contributes to the

release of BTEX compound to the atmosphere. It is also can be used as an intermediates in the synthesis of organic compounds for many consumer products. High concentrations of BTEX can be retrieved from oil and gas operations (Latif, M.T *et al*., 2019), the automotive industry and storage tank filling (Latif, M.T *et al.,* 2019, Ashley L. *et al.,* 2015). In addition, municipal waste, traffic, industrial, plastics, solvent extraction and agriculture effluents are the other important sources (Mingkwan Kitwattanavong et al*.,* 2013). BTEX compound is well known as the compound that are harmful to the environment and human health through various pathways. The exposure to humans especially by direct inhalation may cause adverse effects on human health. The most frequently adverse impacts of BTEX on human health are mainly respiratory irritation and central nervous system damage (A.A.M. Daifullah, B.S. Girgis., 2003). Furthermore, the function and development of the reproductive system, immune system and metabolic system may be affected if there is long-term exposure. It can be seen in several diseases such as aplastic anaemia and acute myelogenous leukemia which is caused by benzene, while exposure to ethylbenzene and xylene was also reported can cause acute eye and skin irritation. BTEX are also reported to have a significant contribution to the formation of oxidants or other air pollutants such as ozone polycyclic aromatic hydrocarbon and ultrafine particles in the atmosphere which may cause serious environmental issues such as haze and ozone depletion (Latif, M.T *et al.,* 2019, Yujie Zhang *et al.,* 2012, Juping You *et al.,* 2018). According to the Department of Occupational Safety and Health Malaysia, based on the Industry Code of Practice 2010, the acceptable limit for total VOCs is 3 ppm. Therefore, the method for BTEX removal has gained increasing attention (DOSH, ICOP IAQ, 2010).

In order to reduce the pollutants, some researchers have conducted the study for controlling the VOC such as adsorption, condensation, photocatalytic oxidation (PCO), negative air ions (NAIs) and non-thermal plasma (NTP) (Das *et al.,* 2004). In recent years, adsorption in bulk separation or purification process has an innovative treatment process in environment application. The adsorption method is effective at low concentration levels which is part per million (ppm). Larger adsorption capacity is achieved by a larger surface area of the filter material and their performance in both equilibrium and kinetics. In this study local sources have come from the agricultural and market were introduced to be converted to activated carbon. The precursor of palm shell, coconut shell, rubber seed shell and bamboo. These materials are abandoned and they can be preferred as raw materials and the price range is also affordable in a market or from the villagers. In preparing the VOCs sampling media, the objective of this study is to verify the sampling media including detection limit, sensitivity, specificity, bias, accuracy, precision, recovery, maximum volume, minimum volume and desorption efficiency. According to Obi et al. (2016), up to 80 % of solid wastes (i.e., 998 million tonnes) per annum are generated from farmyards. In another study, about 337,000 tonnes of agricultural waste was generated in Kuwait while the Abu Dhabi Emirates produced up to 1.2 million tonnes of agricultural wastes in 2019. Developing countries present the worst-case scenario: for instance, Nigeria and India produce up to 200 million and 600 million tonnes (respectively) of agricultural wastes annually with most of the wastes burnt openly in the fields as a disposal attempt (Momoh et al., 2022a; Momoh et al., 2022b). These wastes ranges between fruit peels, shells, hulls, seed pods, bagasse, stalks, leaves. barks, roots, sludges, cobs, pulp, straws, brans, and husks, (Adejumo and Adebiyi, 2020; Ioannou et al., 2015) with the oil palm tree generating the largest share of the agro-wastes in the form of fibres from different parts of the tree (Momoh and Osofero, 2019; Momoh et al., 2020; Momoh et al., 2021) and shells from the extraction of palm kernel (Dungani et al., 2018)

1.2 Flowchart of study

The overall process of the study were described in the flowchart shown in figure 1.2. Begins with the identifications of raw materials and chemical reagent used for the study. The raw materials selection were conducted based on several factors such as the abundancy of the raw materials, price of the raw materials and the sustainability for materials collection. The chemical reagent used for the study was selected based on which chemical can enhance more pore formation towards the carbon during the activation process. Each chemical posses different pore formation towards the carbon during the activation process. Some chemical enhance the microspore formation while others enhance the development of mesopore formation on carbon during activation process. For this study, after conducting several literature reviews on the potential raw materials, palm kernel shell (PKS), coconut shell (CS), rubber seed shell (RSS) and bamboo were selected as the raw materials due to the fulfilment of the criteria set during the early stage of the study. Meanwhile, sodium hydroxide (NaOH) was selected as the chemical reagent for activation process due to the supporting evidence of this chemical which can enchance the formation of macrospore, microspore as well as mesopore on the carbon during the activation process. The production of AC begins with the development and fabrication of the burner for carbonization of the raw materials. The carbonization of each raw materials under certain temperature range between 350- 850^{0}C would convert the raw materials to carbon. Next the carbon will be crush, using the crusher machine to increase the surface exposure before activation process. Proximate analysis was conducted to analysed the ash content, moisture content, volatile content and fixed carbon content. The characteristic of AC produce will be tested using GC-MS for verification of AC produced. Finally the development of solid sorbent tube was conducted for the BTEX detection in IAQ monitoring.

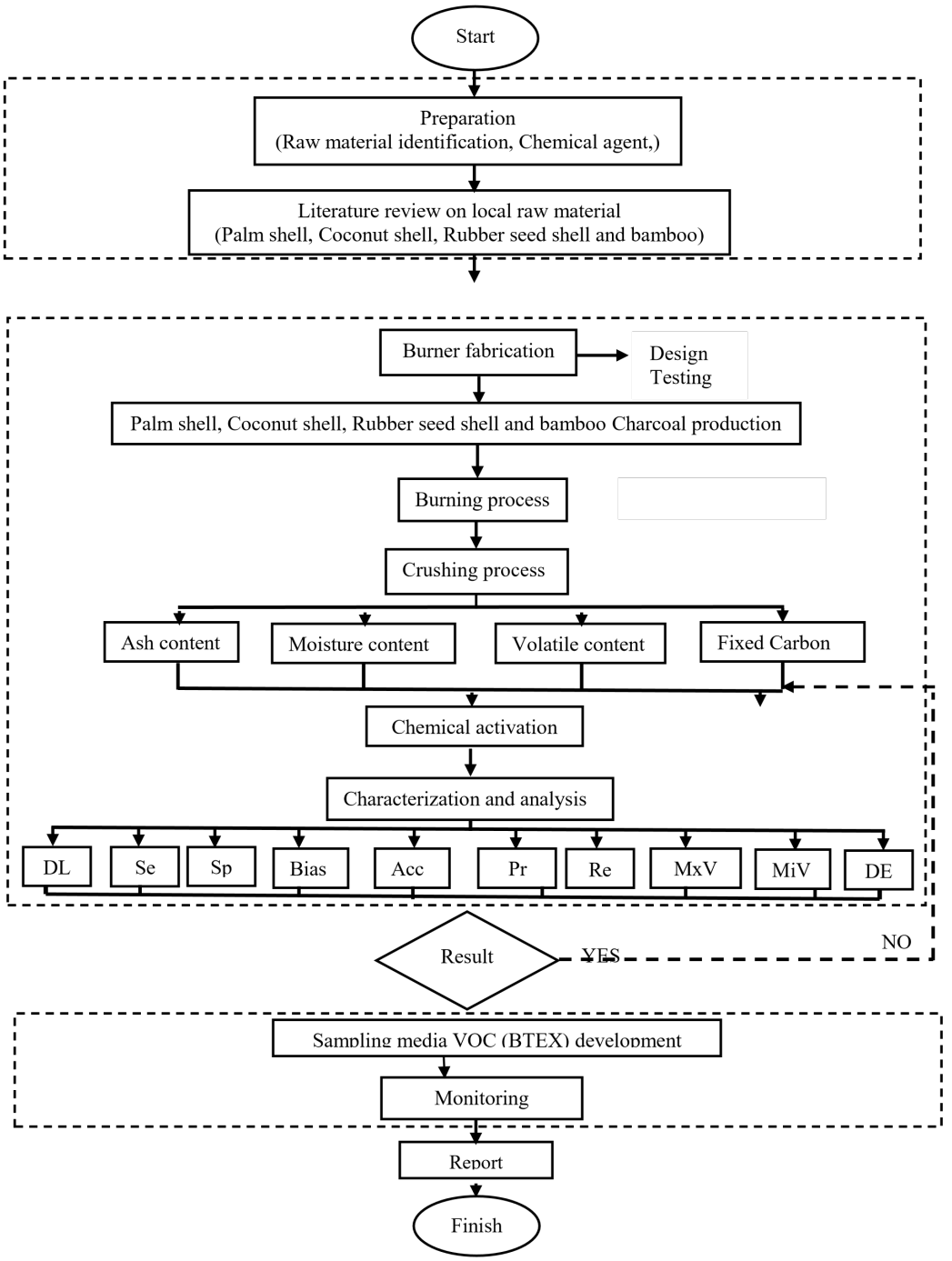


Figure 1.2 : Flow Chart of the Study

2.0 MATERIALS AND METHOD

In this study, the materials that were involved were Palm Shells, Coconut Shells, Rubber Seed Shells and Bamboo. It is collected in the Batu Pahat and Pagoh areas (including from the market and villagers) according to the availability on time respectively. Figure 2.1 a - d shows the raw material of this study. The main focus of this study was on the preparation and characterization of local sources of material to be a sampling medium. The process starts with finding the local source material and burner fabrication (Zakaria, S *et al.,* 2016).

a) Palm Shell b) Coconut Shell c) Rubber Seed Shell d) Bamboo

Figure 2.1 : Raw Material

2.1 Carbonization

The carbonization process of raw palm shells was carried out in the new burner with a carbonizing temperature range 350ºC - 850ºC under an atmospheric environment. The burner was heated directly by a natural gas stove until the final temperature was achieved. The charcoal materials were cooled at room temperature before discharging from the burner. The apparent yield (Y^o) referring to the yield obtained without washing (to remove the residual chemical) was evaluated at this stage using the relation;

$$Y^o(\%) = [W_2 / W_1] \times 100 \quad (2.1)$$

Where,

Y^o = apparent yield, %

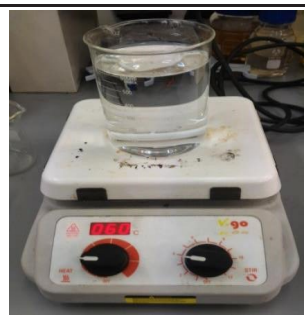
W_1 = weight of impregnated palm shell before carbonization, g

W_2 = weight of palm shell char, g

2.2 Activation

The activation process was conducted by using a furnace at Universiti Tun Hussein Onn Malaysia (UTHM). Palm shell charcoal, which was carbonized with the new burner, was crushed into several sizes using a crusher and sieved with a 0.25 µm - 1 mm particle size. The chemical activation process was conducted by soaking the charcoal in the selected chemical agent (KOH or NaOH) and of known concentration in a crucible. The ratio of palm shell charcoal with chemical solution was 1:1 as shown in Figure 2.2. The chemical agent was dissolved in distilled water, heated at the temperature of 60ºC and stirred for a full mixed electrolyte as demonstrated in Figure 2.2. The palm shell charcoal mixed with KOH electrolyte was soaked for 24 hours and is shown in Figure 2.2. Then, it was washed with water until their pH reached approximately 7 and heated in a furnace at 850ºC with 1 hour holding time in the absence of nitrogen as inert gas to increase the surface area of the samples for adsorption purposes. The activated carbons were later cooled to room temperature. This information is an example and all materials use the same process for activation.

Ratio charcoal and chemical



Chemical solution mixing process

Mix charcoal and electrolyte

Figure 2.2 : Ratio Charcoal and Chemical, Mixing KOH Electrolyte and Mixed Charcoal and Electrolyte

2.3 Proximate analysis

Determination for proximate analysis in AC production was important as the data will show the result for the moisture content, ash content, volatile matter content and the fixed carbon content. There are standards for each parameter with the produced AC must obey for determination of the AC functionality.

2.3.1 Ash content

Total ash is a measure of the mineral oxide content of activated carbon on a weight basis. It is measured by converting the mineral constituents into the respective oxides at 800°C. The ash is mainly composed of silica and aluminium and the amount depends on the base raw material used to produce the product.

2.3.2 Moisture content

Activated carbon is a porous substance that is used in many applications such as water purification, air filters, volatile organic compound (VOC) emission control or metal extraction and recovery. Knowing the moisture content of activated carbon ensures that it does not contain excess water and is within the target moisture content when processed into a finished product. This is especially important if the product can absorb moisture during transport or if it remains exposed in an uncontrolled environment.

2.3.3 Volatile matter

The volatile matter of activated carbon refers to the percentage of coal evaporating organic matter that evaporates after heating for 7 minutes at 900 ℃ under the condition of isolated air. The volatile matter of AC is an indicator of the degree of AC metamorphism, the degree of AC conversion metamorphism Increase, the volatile matter of AC decreases.

2.3.4 Fixed carbon content

Fixed carbon content gives information of the amount of char formation in the thermochemical conversion process. It is the solid combustible residue that remains after the volatiles matter drive off. Higher the fixed carbon, the higher the char production in the thermochemical conversion process as a product yield.

2.4 Analysis

For this project, there has a few analysis should be done according to NIOSH Manual of Analytical Methods (**NMAM**) 1501 (Hydrocarbon, Aromatic) and there are other standards such as NMAM 4000 (Toluene)(Diffusive sampler) and NIOSH method 2549 volatile Organic Compounds (Screening). ASTM D6196 also will be referred to ensure the standard practice for choosing sorbents, sampling parameters and thermal desorption analytical conditions for monitoring VOC in air. To make sure the process is followed step by step, hence the entire sorbent tube production is depicted in figure 2.3.

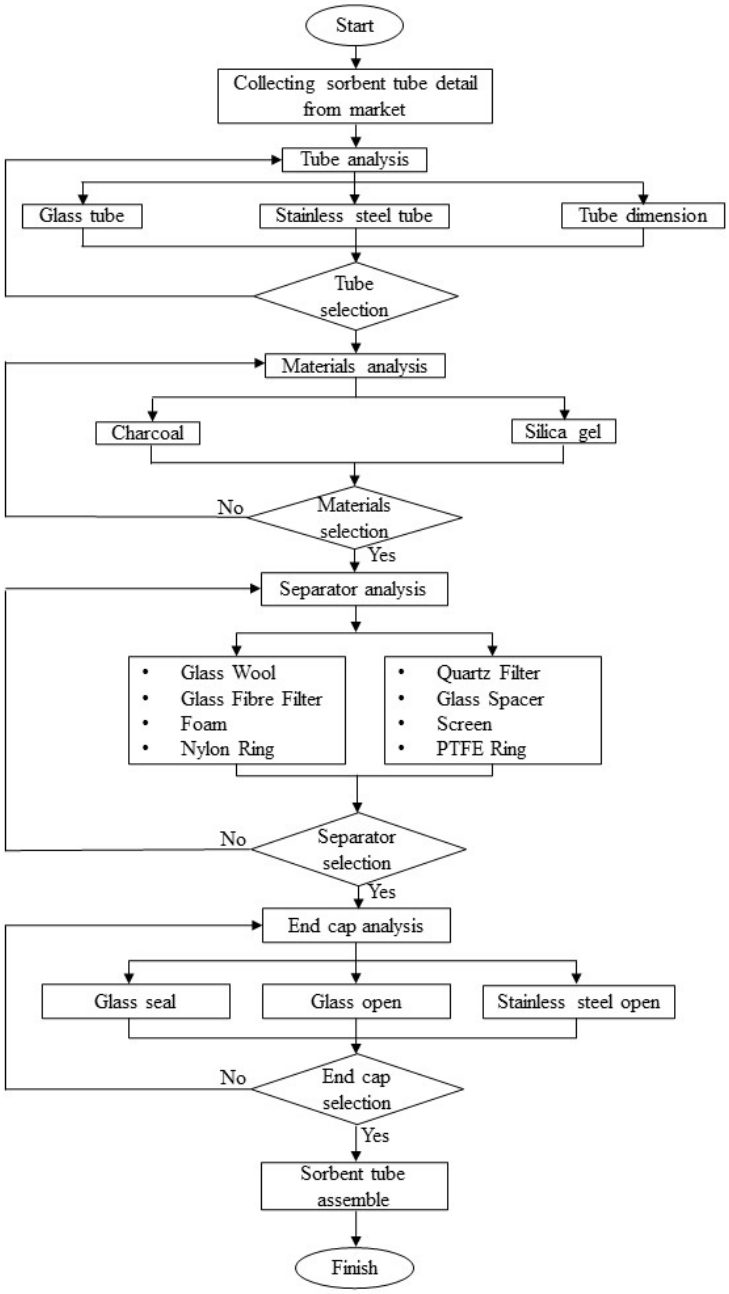


Figure 2.3 : Sorbent Tube Development

The development of solid sorbent tube were discussed as shown in figure 2.3. The process begin with the data collection of commercial solid sorbent tube available in the market. The dimension, materials and process were study before the development process. Next the analysis on the tube for the development process. The existing sorbent tube using glass as the tube, however in this study the tube used was stainless steel tube with dimension of 6 mm X 70 mm compatible with the air sampler tools which required no modification for the usage of the tube in the future. Beside that the advantage of using the stainless steel tube was the tube can be recycled. The materials used as the adsorbent in the market can be either AC or silica gel. And for the study the materials inside the tube was AC which has been produce from PKS, CS, RSS and bamboo. Each sorbent tube available in the market has different separator according to the manufacturer. In the study the separator used for the development of sorbent tube was glass wool separator because of the availability of this materials in

the market and comes with reasonable price. The existing sorbent tube has two method for sealing the tube which were end seal or open end with rubber seal. In the study, the open end with rubber seal were used for the consealing the materials from any contaminant. Stainless steels tube was very hard to seal hence the usage of rubber end cap will provide an althernative solutions towards the problem beside provide an easy access for data collection later.

2.5 Solid Sorbent Tube Preparation

After the verification and validation result from the accredited laboratory has been received, the process of preparation sampling media (solid sorbent tube) is carried out. For this study, the stainless steel tube was chosen because of it availability. Figure 2.4 shows the Diagram of a standard sorbent tube

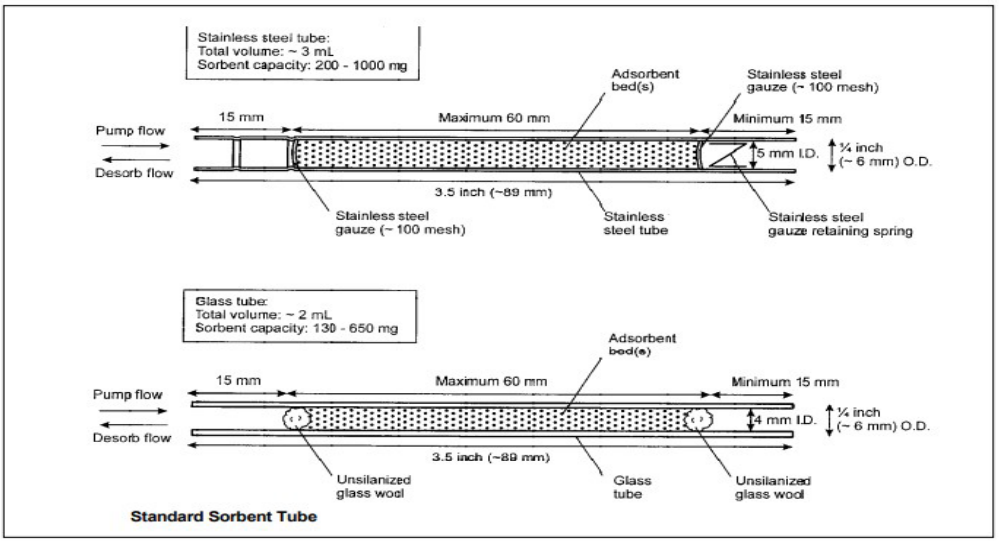


Figure 2.4 : Diagram of a Standard Sorbent Tube (Elizabeth Woolfenden, 1997)

Based on the flow process of sorbent tube development, the tube materials that will be used in the study were glass tubes and stainless-steel tubes with 6 mm outside diameter and 70 mm length. The separator that will be used for the study was cotton glass wool and foam. The end cap for the sorbent tube was open end with a rubber cap. Last but not least the sampling media used for this study come from the activated carbon that has been produced earlier. Table 2.1 shows the materials used for the study. The process of assembling the sorbent tube was conducted in a clean room to prevent contaminants adsorbed by the AC. The process begins with measuring the amount of sampling media. For this study, according to the manufacture, the weight of the AC was 100 mg on one side and another 50 mg of AC on the other side. To get the accurate measurement the weighing process was using the digital balance with 0.001 g accuracy. Table 2.1 shows the materials used for the assembly of the sorbent tube.

Table 2.1 Materials Used for the sorbent tube development

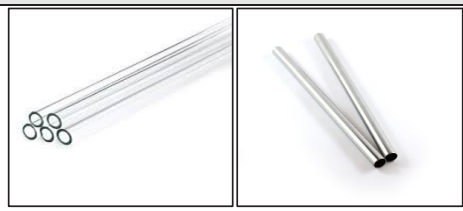
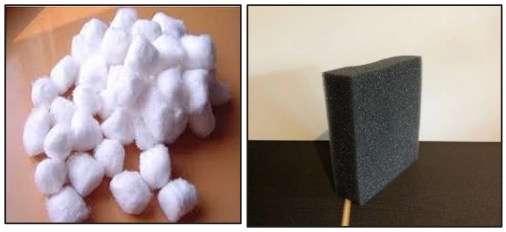
No	Image	Materials
1		Glass tube and stainless-steel tube with dimension of 6mm OD and 70mm length
2		Separator inside the tube using the cotton glass wool and foam
3		Rubber end cap for sealing the sampling media from contaminant
4		AC from earlier production from PKSAC, CSAC, RSSAC, and BAC

Figure 2.5 shows the sample of sampling media that has been developed by the research team. The sampling media has been developed in two packages such as solid sorbent tube (glass) and solid sorbent tube (stainless steel). Based on the diagram of the standard sorbent tube, the team has decided to use a stainless steel tube and survey the availability and price after making some comparisons with the glass tube.

a) Solid sorbent tube (glass) **b) Solid sorbent tube (stainless steel)**

Figure 2.5 : Sample of Solid Sorbent Tube of Sampling Media

3.0 RESULT

3.1 Proximate analysis of AC

Proximate analysis is a way to determine the distribution of products when the samples are heated under specified conditions. Proximate analysis is one of the first analyses performed on coal after mining. The test involves heating the coal under various conditions for variable amounts of time to determine moisture, volatile matter, fixed carbon, and ash yield. Moisture in coal is determined by heating the coal to a temperature slightly above the boiling point of water (377–383K) and holding it at that temperature until there is no further weight loss. Volatiles are measured based on additional weight loss by heating the coal to 950°C 1223K in a N_2 atmosphere. The leftover residue, known as char, is burnt in O_2 to measure the incombustible residue called ash. Fixed carbon is the organic matter in the char, determined by the difference between 100 and the sum of the percentages of volatile matter, ash, and moisture.

One of the most important parameters obtained from proximate analysis is the moisture content. A higher moisture content in the coals tends to reduce the gasifier temperature and consequently the rate of conversion of carbon during gasification (Schobert, 1995). Moreover, moisture can also affect the way the coal is fed into the gasifier. When the inherent moisture increases, the pulverized particles in the dry feed system tend to agglomerate, resulting in unsteady feeding of the coal. Based on a study conducted with a KBR transport gasifier, inherent moisture of greater than 28% for a particle size distribution between 200–800 μm can result in an unsteady operation of lock hopper feeders (Dorminey *et al*., 2009). For finer feed particles fed into entrained-flow gasifiers, a guideline of less than 2 wt% moisture is recommended. The limit on moisture is to ensure acceptable pneumatic conveying characteristics of coal (Yun *et al*., 2007). Besides affecting the dry feeding system, high-moisture coals can also affect the overall efficiency of slurry-feed gasifiers. High-moisture coals are typically low-rank coals, which are of low calorific value. Feeding such coals as slurry with added water can affect the overall energy density of the slurry, leading to lower efficiency of the gasifier.

Another parameter in the proximate analysis is ash. It is important to recognize that coal has mineral matter and not ash. Ash is the product of combustion or gasification (high temperature oxidation or reduction of mineral matter). Coals with high ash yield are not preferred, as a large feed rate is needed to generate the same amount of energy (Mastalerz *et al*., 2008). Additionally, the sensible heat available for gasification reactions is reduced in the presence of a substantial amount of ash in the gasifier, resulting in reduced efficiency. Besides the loss of sensible heat, melt phase derived from ash can cover char structure, leading to a higher amount of unconverted carbon in the slag. Mastalerz *et al*. (2008) reported that the maximum ash yield from coal that fixed-bed, fluidized-bed, and entrained-flow gasifiers can handle are 15%, 40%, and 25%, respectively.

The other parameter that is determined is the volatile matter. Higher volatile matter in coals is advantageous because it is evolved as gas instantaneously, leaving behind a small amount of char with higher porosity (Mastalerz *et al*., 2008). The rate of conversion of char to gas phase is much slower, and therefore a lesser amount of char in the gasifier results in higher gasification efficiency. The amount of volatile matter in coal decreases with increasing rank. Table 3.1 indicated the summary of proximate analysis in this study.

Table 3.1 Proximate Analysis in This Study

Material	Current study			
	Moisture content	Ash content	Volatile matter content	Fixed carbon content
PKSAC	3.56	2.56	10.81	83.07
CSAC	4.26	0.79	12.45	82.50
RSSAC	4.55	5.48	8.67	81.31
BAC	5.27	7.22	10.64	76.86
	Quality standard			
	Max 15%	Max 10%	Max 25%	Min 65%

3.2 Characteristics of Charcoal

The characteristics of palm shell charcoal are important and to be examined before activated carbon production to produce excellent and high quality products. Among the characteristics considered for charcoal production are the percentage of

moisture content, ash content, volatile content and carbon fixed before the production of activated carbon. Figure 3.1 a – d shows the charcoal of each raw material respectively.

Figure 3.1 (a) : Palm Shell Charcoal

Figure 3.1 (b) : Coconut Shell Charcoal

Figure 3.1 (c) : Rubber Seed Shell Charcoal

Figure 3.1 (d) : Bamboo Charcoal

The testing based on NMAM and NOSH methods by using Gas chromatography-mass spectrometry (GC-MS) will be discussed further. Figure 5.1 shows the GC-MS at the Faculty of Engineering Technology, Universiti Tun Hussein Onn Malaysia (UTHM) where the testing was conducted to ensure the validation and verification data based on NMAM 1501. The sampling media has been sent to an accredited laboratory for validation and verification for BTEX absorbent.

3.3 Result Validation and Verification

Result validation and verification of Palm Shell Activated Carbon has been conducted at the accredited laboratory to ensure the valid data will be represented especially some of the evaluation of the benzene, toluene, Ethyl-benzene and Xylene. The summary of the validation data for Palm Shell Charcoal Test for BTEX absorbent has been tabulated in table 3.2. Meanwhile table 3.3, 3.4 and 3.5 show the summary of the validation data for Coconut Shell Charcoal, Rubber Seed Shell Charcoal and Bamboo Charcoal Test for BTEX.

Table 3.2 Summary of Validation Data For Palm Shell Charcoal Test For BTEX Absorbent.

No.	Parameter	Benzene	Toluene	Et-Benzene	M/P -Xylene	O-Xylene
(i)	Limit of Detection	0.10	0.05	0.11	0.07	0.10
(ii)	Sensitivity	0.34	0.16	0.35	0.24	0.32
(iii)	Specificity	87.5	87.5	100	100	91.6
(iv)	Bias	11.027	11.021	11.126	11.146	11.206
(v)	Accuracy (± %)	0.092	0.082	0.079	0.071	0.087
(vi)	Precision	80.60	80.98	66.09	68.70	62.76
(vii)	% Recovery	80.60	80.90	66.09	68.70	62.76
(viii)	Maximum Volume	30	8	24	23	23
(ix)	Minimum Volume	5	1	1	2	2
(x)	Desorption Efficiency					
	0.5 µg	0.614	0.761	0.687	0.629	0.503
	1.0 µg 2.5 µg	1.030	1.050	0.575	0.578	0.524
	5.0 µg	0.905	0.952	0.654	0.714	0.689
	15.0 µg 30.0 µg	1.045	0.991	0.730	0.820	0.782
		0.961	0.787	0.617	0.688	0.658
		0.759	0.677	0.565	0.613	0.598
Note	Range 0.2 to 30 µg	**Good**	**Good**	**Acceptable**	**Acceptable**	**Acceptable**

Table 3.3 Summary of Validation Data of Coconut Shell Charcoal Test For BTEX Absorbent

No.	Parameter	Benzene	Toluene	Et-Benzene	M/P -Xylene	O-Xylene
(i)	Limit of Detection	0.11	0.12	0.08	0.12	0.08
(ii)	Sensitivity	0.35	0.37	0.24	0.38	0.26
(iii)	Specificity	75	75	54.1	54.1	0.5
(iv)	Bias	10.871	10.876	10.412	10.825	10.814
(v)	Accuracy (±%)	4.77	4.28	6.88	3.82	5.93
(vi)	Precision	0.063	0.070	0.057	0.064	0.054
(vii)	% Recovery	96.17	95.90	100	100	100
(viii)	Maximum Volume	30	8	24	23	23
(ix)	Minimum Volume	5	1	1	2	2
(x)	Desorption Efficiency					
	0.5 µg	0.906	0.928	1.078	1.006	1.043
	1.0 µg 2.5 µg	0.959	0.996	1.035	0.985	1.095
	5.0 µg	1.093	1.076	1.124	1.115	1.121
	15.0 µg 30.0 µg	1.014	1.003	1.029	1.053	1.049
		0.975	0.998	0.924	0.972	0.935
		0.965	0.946	0.907	0.945	0.914
Note	Range 0.2 to 30 µg	**Excellent**	**Excellent**	**Excellent**	**Excellent**	**Excellent**

Table 3.4 Summary of Validation Data of Rubber Seed Shell Charcoal Test For BTEX Absorbent

No.	Parameter	Benzene	Toluene	Et-Benzene	M/P -Xylene	O-Xylene
(i)	Limit of Detection	0.11	0.08	0.16	0.07	0.09
(ii)	Sensitivity	0.37	0.24	0.51	0.24	0.29
(iii)	Specificity	95.8	100	95.8	95.8	100
(iv)	Bias	10.946	10.939	11.193	10.941	10.976
(v)	Accuracy (±%)	11.26	10.79	9.92	10.83	13.58
(vi)	Precision	0.060	0.048	0.086	0.061	0.058
(vii)	% Recovery	88.74	89.21	90.08	89.17	86.42
(viii)	Maximum Volume	30	8	24	23	23
(ix)	Minimum Volume	5	1	1	2	2
(x)	Desorption Efficiency					
	0.5 µg	0.806	0.839	0.865	0.944	0.903
	1.0 µg					
	2.5 µg	0.886	0.895	0.888	0.900	0.934
	5.0 µg	0.930	0.891	0.913	0.828	0.770
	15.0 µg	0.886	0.919	0.881	0.867	0.769
	30.0 µg	0.927	0.947	0.924	0.903	0.920
		0.965	0.946	0.907	0.945	0.914
Note	Range 0.2 to 30 µg	**Very Good**	**Very Good**	**Very Good**	**Very Good**	**Very Good**

Table 3.5 Summary of Validation Data of Bamboo Charcoal Test For BTEX Absorbent

No.	Parameter	Benzene	Toluene	Et-Benzene	M/P -Xylene	O-Xylene
(i)	Limit of Detection	0.20	0.22	0.13	0.22	0.25
(ii)	Sensitivity	0.64	0.73	0.41	0.73	0.81
(iii)	Specificity	83.3	83.3	100	100	100
(iv)	Bias	11.027	11.060	11.178	11.110	11.088
(v)	Accuracy (±%)	30.33	22.72	34.54	27.71	25.29
(vi)	Precision	0.092	0.165	0.126	0.147	0.142
(vii)	% Recovery	69.67	77.28	65.46	72.29	74.71
(viii)	Maximum Volume	30	8	24	23	23
(ix)	Minimum Volume	5	1	1	2	2
(x)	Desorption Efficiency					
	0.5 µg	0.679	0.873	0.578	0.753	0.899
	1.0 µg	0.993	1.134	0.449	0.609	0.629
	2.5 µg	0.809	0.903	0.359	0.747	0.705
	5.0 µg	0.721	0.758	0.692	0.724	0.693
	15.0 µg	0.716	0.707	0.666	0.674	0.623
	30.0 µg	0.691	0.688	0.693	0.692	0.649
Note	Range 0.2 to 30 µg	**Acceptable**	**Acceptable**	**Acceptable**	**Acceptable**	**Acceptable**

4.0 DISCUSSION

Analysis that has been conducted at Accredited Laboratory for the Palm Shell Charcoal, Coconut Shell Charcoal, Rubber Seed Shell Charcoal and Bamboo Charcoal was summaries in Table 3.1 to table 3.4 respectively. Based on the table 3.1, the result of Palm Shell Charcoal shows the limit of detection (µg/sample), the limit for reporting (µg/sample desorption efficiency range tested from 0,5 µg – 30.0 µg, % recovery, accuracy and precision). The result was excellent for all tested data since the element has fulfilled the standard based on NIOSH Manual of Analytical Methods (NMAM) 1501. At this point, the palm shell activated carbon is the second best if compared with the other local materials in this study (Coconut Shell, Rubber Seed Shell and Bamboo). The idea in the earlier stage (proposal) strongly recommended of using Palm Shell it is because of the volume of Palm shells available in the market. At the same time, the proposal takes into account the potential of palm shell since it has a high porosity and large absorption capacity, especially for chemicals such as BTEX. Meantime, the potential of shell activated carbon to be a sampling media is rated as good for benzene and toluene absorption and acceptable for ethyl-benzene and xylene. Table 4.1 shows the GM-MS testing on charcoal sorbent tubes.

Table 4.1 GC-MS testing for charcoal sorbent tube (Ashouri *et al.*, 2020)

Air sample*	GC–MS analysis (mg in liter air)	Benzene added (Conc./time)[b]	Found ST-SPE (mg in liter air)[a]	Recovery (%)
Sample 1	0.25 ± 0.01	–	0.24 ± 0.01	96.0
		0.3/1	0.53 ± 0.03	96.7
Sample 2	0.84 ± 0.01	–	0.85 ± 0.04	101.2
		0.9/3	1.73 ± 0.08	97.7
Sample 3	1.12 ± 0.02	–	1.09 ± 0.05	97.3
		1.2/4	2.27 ± 0.11	98.3
Sample 4	2.36 ± 0.04	–	2.39 ± 0.12	101.3
		2.4/8	4.81 ± 0.23	100.8
Sample 5	3.17 ± 0.05	–	3.16 ± 0.14	99.6
		3.0/10	6.12 ± 0.29	98.7

The result of Coconut Shell Charcoal shows that the limit of detection (µg/sample), Limit for Reporting (µg/sample desorption efficiency range tested from 0,5 µg – 30.0 µg, % recovery, accuracy and precision). The result for all tested data since the element has fulfilled the standard based on NIOSH Manual of Analytical Methods (NMAM) 1501. At this point, the coconut shell activated carbon is the best if compared with the other local material. The sampling media for VOC absorption using coconut shell activated carbon is highly recommended since all sample tests show excellent evaluation parameters for benzene, toluene, ethyl-benzene and xylene. That reason why, the company that produces solid sorbent tubes were select coconut activated carbon (normally known as coconut charcoal) as a sampling media that has been established in the occupational hygiene market for BTEX absorption. Table 4.2 shows the sample preparation for BTEX adsorption carbon fibre sorbent tube

Table 4.2 Electrothermaly Conditioned Carbon Fibre Sorbent Tube on BTEX (Pekiyi et al., 2023)

Parameter	Benzene	Toluene	Ethylbenzene	o-xylene	p-xylene
ConditioningCurrent / Time			9.5 A / 120 s		
Flow Rate(mL min $^{-1}$)			20–200		
Sample Volume ∗ (L)	≤ 1.0	≤ 1.5	≤ 2.0	≤ 2.0	≤ 2.0
Relative Humidity(%)	≤ 70	≤ 100	≤ 100	≤ 100	≤ 100
Sampling Temperature(°C)	10- 30	10–30	10–50	10–50	10–50
Calibration Equation	y = 4.1543x + 1.3243	y = 5.0733x + 1.6526	y = 6.2304x - 0.2571	y = 5.4868x + 4.2136	y = 5.6930x + 3.5914
R 2	0.9878	0.9979	0.9999	0.9925	0.9955
Standard ErrorSlop / Intercept	0.2665 / 1.1550	0.1166 / 1.1122	0.0369 / 0.3513	0.2133 / 3.9343	0.1718 / 3.2402
LOD/ LOQ(mg m $^{-3}$)	0.11/0.36	0.09/0.29	0.07/0.24	0.08/0.27	0.08/0.26
∗∗Intraday Repeatibilitiy (%)	1.3	1.3	1.1	1.1	1.8
∗∗Interday Reproducibility (%)	6.9	5.6	8.6	9.5	9.3
Linear Range (mg/m ^{3})	0.36 – 8.35	0.29 – 21.2	0.24 – 21.2	0.27 – 42.8	0.26 – 43.7

∗ Concentration of each analyte in gas sample was 4.9 mg m $^{-3}$.

Meanwhile, table 3.3, shows the result of Rubber Seed Shell Charcoal that the limit of detection (µg/sample), limit for reporting (µg/sample desorption efficiency range tested from 0,5 µg – 30.0 µg, % recovery, accuracy and precision. The result was rated as good for all tested data since the element has fulfilled the standard based on the NIOSH Manual of Analytical Methods (NMAM) 1501. At this point, the rubber seed shell activated carbon is the second best if compared with the other local material. The idea in the earlier stage (proposal) recommends using a rubber seed shell since the shell has high porosity and the size is also suitable for carbonization. At the same time, the proposal takes into account the potential of rubber shell since it has been used for the water treatment process and large absorption capacity, especially for heavy metal /substance. Meantime, the potential of rubber shell activated carbon to be a sampling media is rated as very good for benzene, toluene, ethyl-benzene and xylene absorption. This study shows that an alternative material is available to replace the coconut shell and the other material selected is Bamboo Charcoal. Table 4.3 shows the LOD of BTEX using a charcoal sorbent tube

Table 4.3 Limit of Detection For BTEX (Soury *et al.*, 2022)

Analyte	Repeatability, mg/m^3 RSD % N = 6 Injections		Reproducibility, mg/m^3 RSD% N = 6 injections		LOD, mg/m^3	LOQ, mg/m^3	LDR, mg/m^3
Benzene	0.3 1.6 8	6.4 7.5 7.9	NTD1 NTD2 NTD3	8.1 10.1 6.2	0.16	0.52	0.2–22
Toluene	15 75 377	7.3 8.3 5.7	NTD1 NTD2 NTD3	6.3 8 10.6	0.38	1.1	0.4–380
Ethybenzene	17 87 434	5.5 6.6 9.4	NTD1 NTD2 NTD3	5.3 9.4 11	0.5	1.4	0.5–435
m-, *p*-Xylene	87 434 2171	7.4 6.3 8.2	NTD1 NTD2 NTD3	8.1 12.3 9.4	0.4	1.32	0.3–2200
o-Xylene	87 434 2171	8.6 13.2 10.1	NTD1 NTD2 NTD3	9.7 8.9 12.3	0.4	1.41	0.4–2200

The result of Bamboo Charcoal shows that the limit of detection (µg/sample), limit for reporting (µg/sample desorption efficiency range tested from 0,5 µg – 30.0 µg, % recovery, accuracy and precision. The result was rated as good

for all tested data since the element has fulfilled the standard based on NIOSH Manual of Analytical Methods (NMAM) 1501. At this point, the bamboo activated carbon is in the last position if compared with the other local materials in this study (coconut shell, rubber seed shell and palm shell). The idea in the earlier stage (proposal) recommends of using bamboo since the availability and the size are also suitable for carbonization. At the same time, the proposal takes into account the potential of bamboo since it has been highlighted as a new commodity by the Ministry of Plantation Industries and Commodities Malaysia (MPIC). Table 4.4 shows several comparisons of sorbent type.

Table 4.4 Comparison of sorbent type adsorption performance (Pekiyi et al., 2023)

Parameters	References				
	Mohammadi et al., 2017	**Gallego-Díez et al., 2016**	**Bocchini et al., 2009**	**Hajizadeh et al., 2018**	**Pekiyi et al., 2023**
Analysis Method	GC–MS	GC-FID	GC-FID	GC-FID	GC-FID
Sampling Method	NTD **	Passive sampling	SPME ***	Adsorption Tube	Adsorption Tube
Sorbent Type	Carbotrap B	Activated charcoal	Carboxen-Polydimethylsiloxane	Charcoal	Electrotermal Conditioned CF
BTEX Sorption Capacity (μg g^{-1})	NA *	NA *	NA *	NA *	3.18
LOD (mg m^{-3})	0.03–0.04	47–65	0.01–0.02	0.7–1.7	0.07–0.11
Linear Range (mg m^{-3})	0.03–25	300–97.000	0.01–4.3	2.3–218	0.24–45
Sampling Volume (L)	0.005	NA *	Optimization at 2 L 50 m^3 < Sample Volume	24	1
Analysis Time	32 min	28 days	5 h	3 h	25 min
Recovery (%)	95.3–99	75.0 - 98.2	NA *	NA *	95.5–102.7

* Not available.
** Needle trap device.
*** Solid phase micro extraction.

5.0 CONCLUSION

The four local source materials such as Palm Shell, Coconut Shell, Rubber Seed Shell and Bamboo previously known as agricultural waste could be converted to raw materials for solid sorbent tubes. Based on this study, it is shown that the best raw material is coconut shell followed by Rubber Seed Shell. Meanwhile, Palm Shell and Bamboo is rated third place and fourth place respectively. The potential of raw material from local sources to be an activated carbon for VOC absorption is huge, hence the support from the government agencies and related party need to be enhanced for the betterment of society in Safety and Health knowledge dissemination. On the other hand, the result obtained from the verification analysis and validation from the accredited laboratory, the four raw materials said above from the local source (agricultural waste / local sources) could be used as activated carbon for solid sorbent tube for VOCs detection especially on Benzene, Toluene, Ethylbenzene and Xylene (BTEX).

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