Applied Chemistry Project

Project title Development of bismuth-doped amorphous cellulose for X-ray shielding

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Program Bachelor of Science in Applied Chemistry

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Development of bismuth-doped amorphous cellulose for X-ray shielding

by
Jinyada Nikhong

In Partial Fulfillment for the Degree of Bachelor of Science Program in Applied Chemistry (International Program)
Department of Chemistry, Faculty of Science
Chulalongkorn University
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Student Name Miss Jinyada Nikhong Student ID 6033802123
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Abstract

Nowadays, there are various types of x-ray applications in human daily life. The uses of radiation required proper shielding equipment to prevent overexposure of radiation. Shielding material made from $BiPO_4$-AC from used paper were investigated in this study. Three different ratio of $BiPO_4$/amorphous cellulose were prepared and tested using different ratio of silicone rubber (SR) as fabric coating. Radiation attenuation ratio (%RAR) were measured at 30, 40 and 60kV. The result showed that 1:1 $BiPO_4$-AC with 1:4 filler-SR performed the best shielding performance at 35.23% at 30kV with the presence of 0.569 g of $BiPO_4$. The increased of $BiPO_4$ content also resulted in the increase of shielding efficiency of the sample. Furthermore, $BiPO_4$/AC substance with higher ratio shown better binding texture. Hence, the addition of amorphous cellulose could enhance the strength of the $BiPO_4$/AC composited compound which give them ability to be further developed into different form of non-toxic x-ray protection equipment.

Keyword : Bismuth Phosphate, amorphous cellulose, X-ray shielding, fabric coating

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Table of Content

Chapter 1
Introduction

1.1 Introduction to the research problem and significance

There are various applications of X-radiation in several branches, including everyday use and industrial work. X-ray is used to locate and inspect the chemical characteristics of an artefact in archaeological science, a cancer treatment tool for radiotherapy, or baggage scanning in airport security system. In the medical field, X-ray has been used to diagnose the abnormal condition of patients specific organs, as well as halting and killing tumor cells for over one hundred years.[1] However, there are severe health hazards that can be caused as side effects from radiation exposure.

Effects on human health from such ionizing radiation can be divided into two types; stochastic effect and deterministic effect. People who are in contact with radiation for a long period of time even with low amount, such as nuclear plant workers, doctors, or patients who receive continuous cancer treatment has high risk of having cancers, or second cancers after treatment, as stochastic effect. On the other hand, deterministic effect, which is an instant effect from receiving large amount of radiation in short time can cause skin irradiation, hair loss, necrosis or cataract.[2] X-ray is a high energy ray that can affect not only on cancers cell, but also on healthy issues around the area. Thus, radiation protection are essential either for a planned exposure (treatment) or an emergency exposure.

There are 3 main factors that control the amount of radiation; time, distance, and shielding. As well as the term 'ALARA', which stands for 'As Low As Reasonably Achievable'. is the common principle for all practices that include radiation. The amount of time taken, the distance between the radiation source and patients or workers and the amount of dose required in each specific work needs to be calculate to be as low as possible in order to ensure the safest working environment with the most effective result. Apart from that, radiation shielding equipment has to be apply at all time for both workers and patients to prevent them from an uncontrollable situation, as well as reducing the risk of the side effect from radiotherapy.

Each type of radiation interacts differently with the matrices. X-ray, which has high penetrating power is able to pass through objects except the one with high atomic number. Considering high density and high atomic number properties in order to have a good shielding performance, lead became the main material chosen to make radiation shielding equipments due to its properties as well as its cost effectiveness. Lead-composite materials such as lead garment, lead sheet, lead gloves and glasses are commonly used to protect workers from radiation. However, due to its high weight and inflexibility, even the composited one, lead shielding equipment can cause injuries to users when wearing them for a long period of time. Moreover, lead is a toxic metal.[3] It requires special disposal treatment of hazardous deteriorated lead-containing equipment, which could be extremely unfriendly to the environment.

There have been several attempts regarding to the development of non-lead radiation protective gears. Since the compound's atomic number affects directly to the shielding efficiency of the equipment, other heavy metals have been investigated as alternative choices. Bismuth, showing good result as an X-ray shielding contributor due to its high-Z and density, high melting point, low conductivity but yet non toxic.[4] Bismuth is also available in powder form which will increase its dispersibility when combining with other binding material, leading to even better shielding performance.

Cellulose is the most abundant natural biopolymer that consists in human everyday use for over thousand of years. It exists in all plants and in various forms such as cotton, bandage, textiles, as well as paper we are using. Cellulose consists of 2 regions; crystalline and amorphous.[5] Previous studies indicate that amorphous cellulose can conduct faster and better chemical modification, having high absorption ability and is able to immobilize functional group-containing substances.[6] In this study, amorphous cellulose prepared by phosphoric acid were chosen as a polymer matrix that will reinforce our main absorber, bismuth, to disperse evenly in other binding component which can be develop further into various types of shielding equipment.

According to the environmental and health concerns as a result of using lead shielding, this study aim to look into the development of lead alternative material made from bismuth phosphate/amorphous cellulose by the regeneration of Bismuth nitrate solution and cellulose in phosphoric acid. This composited substance can be further apply to various types of shielding material such as coated fabric, radiation shielding rubber sheet, or nano coating spray which is flexible, light, sustainable and non toxic to both human and environment.

1.2 Research objectives

1. To develop environmental friendly, light weight, and sustainable shielding material from Bismuth and amorphous cellulose as alternative to toxic lead by studying the appropriate condition for cellulose preparation, acid hydrolysis and appropriate ratio of Bismuth powder in cellulose.
2. To investigate the chemical composition of $BiPO_4$/amorphous cellulose.
3. To examine the shielding ability of $BiSO_4$/amorphous cellulose.

1.3 Literature search

1.3.1 X-radiation. [1], [7]

X-radiation or x-ray is one type of ionizing radiation that was discovered by German physicist Wilhelm Conrad Röntgen in 1895. There are two mechanism of how x-ray is produced. The first one states that x-ray occurs when there is a change in acceleration of electron when the electron beam is entering electric field of the nucleus, the electron then slow down and being deflected and emit x-radiation photon. This process is called bremsstrahlung or braking radiation. The second one indicates that x-ray can be generate from the change of electron from higher energy level to lower energy level, or in other words, from outer orbit to inner orbit. When the electron beam hit the metal target, the electron in the inner shell got removed, then the outer level which has higher energy fill in the space left by the the first electron. This caused excess energy and it then got release in form of x-ray. This process is called characteristic x-ray.

X-rays in use in recent days are produced in a vacuum x-ray tube. Electrons are being accelerated from heated cathode direct to the metal anode (i.e tungsten). After electrons strikes the metal anode, both type of x-ray; braking and characteristic, can be produced within the tube by the mechanism mentioned earlier. Accelerating energy of electrons and the type of metal anode depends on the usage purpose.

1.3.2 Interaction of X-rays with matters. [9]

When x-rays are produced, each photon has unequal energy. High energy photon has enough power to transmit through matters while the low energy one could induce photon scattering and energy absorption, which are the main concept of how x-ray shielding works. There are 3 types of interactions between X-ray and matter that are related to shielding mechanism; Photoelectric effects, Compton scattering effect and Reyleigh scattering effect.

1.3.2.1 Photoelectric effect

Photoelectric effect occurs when photon strikes the inner shell of an atom of the matrix and induced the removal of the electron when the binding energy of that electron is less that the energy of photon. When that happens, the energy of the photon will be completely absorbed so no radiation can pass through the matrix. This effect is proportional to the atomic number of the medium, since the higher Z metal will have higher energy at the inner shell, so in order for the electron to be removed, all of the photon energy has to be completely transferred to the electron.

1.3.2.2 Compton scattering

Compton scattering occurs with loosely bounded electron in the outer shell and low energy photon. When photon hits the electron, the photon change its direction and partial energy are transfer to that electron. This effect is proportional to the electron density and the medium density.

1.3.2.3 Rayleigh scattering

Rayleigh scattering occurs when low energy photon interact with electron of high-Z atom. The photon will be deflected without transferring its energy, only the direction will be changed.

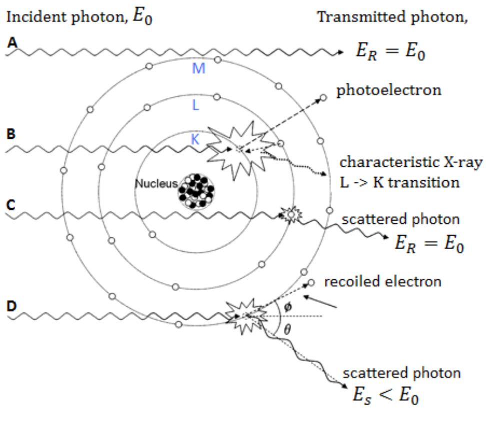


Figure 1-1 Interaction of x-ray with electrons A) No interaction B) Photoelectric effect C) Rayleigh scattering D) Compton scattering

1.3.3 X-ray attenuation [10]

X-ray attenuation can occur after the interaction of radiation beam with matters. How much energy were attenuate is dependent on the initial intensity of photon, density (Mass attenuation coefficient), thickness (Linear attenuation coefficient) and atomic number of matrices. Higher Z tend to have better shielding performance due to its high number of electrons, meaning and high bounding energy. X-ray attenuation efficiency can be obtained from the Linear attenuation coefficient (μ) equation:

$$I = I_0 e^{-\mu x} \quad (1)$$

$$\mu = \frac{In\left(\frac{I}{I_0}\right)}{t} \quad (2)$$

Where *I* is the x-ray intensity after passing through shielding material, I_0 is the initial x-ray intensity before passing through shielding material, *x* is the thickness of the shielding material (cm), while μ can be calculate from equation (2). Radiation attenuation ratios (%RAR) can be calculate from equation (3).

$$\%RAR = \frac{I_0 - I}{I_0} \times 100 \quad (3)$$

1.3.4 Bismuth for X-ray shielding [11]

Bismuth is a non toxic heavy metal with atomic number of 83, hence it has an ability to shield radiation. There were past studies that has investigated the shielding ability of Bismuth compounds such as Bi_2O_3, BiI_3 or Bi powder with polymer. Nabahat et al. [8] studied the shielding efficiency of Bi powder with silicone rubber (SR) for textile coating comparing to Tungsten and Barium Sulphate. The result has shown that Bi-SR has the best shielding performance of over 90% attenuation ratios at 100kV. Although there are still no related studies that use Bismuth phosphate as an radiation absorber yet, $BiPO_4$ has very low solubility, thus it is able to apply into different type of matrices such as polymer composited material or coating emulsion.

1.3.5 Amorphous cellulose

Cellulose is a polysaccharide that contain long chain of β-1,4-D-glucose molecules. According to its polysaccharide structure, there are large amount of hydroxyl group that can form network of hydrogen bonding exist along the cellulose, result in the packed arrangement of elementary fibril of crystalline region and amorphous region. Amorphous region is the area where the hydroxyl group along the chain arranged irregularly, which make the reaction take place easier than the crystalline region.[12]

Cellulose exists in most of plants cell walls which make them become the most abundant natural polymer in the world. According to its properties of having low density, high surface area and are able to undergo chemical and physical modification, cellulose were chosen to be a polymer matrix that would help our absorber, Bi, disperse better in any form of applications.

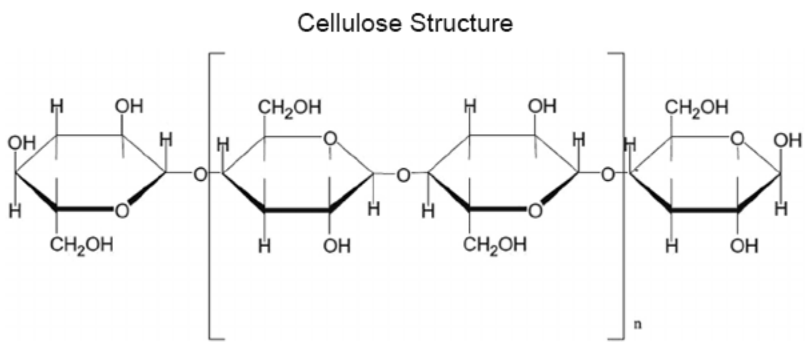


Figure 1-2 Chemical structure of cellulose

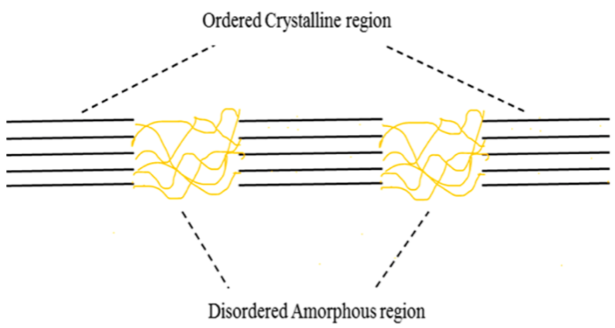


Figure 1-3 Crystalline and Amorphous region of cellulose

Chapter 2
Experimental

2.1 List of equipment and instrument

1. 1L beaker
2. 500ML beaker
3. 200ML beaker
4. 100ML beaker
5. Stirring rod
6. Stainless spatula
7. Dispensing spoon
8. Litmus paper
9. Blender
10. Wash bottle
11. Dropper
12. Laboratory pot
13. 500ML glass bottle
14. Vacuum filter
15. Stainless tray
16. Oven
17. Freezer
18. High speed homogenizer
19. Centrifuge
20. Clear plastic pad
21. 100% cotton fabric
22. Paper tape
23. Stainless ruler
24. Clear plastic sheet frame

2.2 List of chemicals and materials

1. Bismuth Nitrate
2. Used brown envelope
3. Sodium Hydroxide
4. Hydrogen Peroxide
5. Distilled water
6. Phosphoric acid
7. Nitric acid
8. RTV silicone rubber

2.3 Experimental procedure

2.3.1 Preparation of cellulose

100 g of envelope paper was cut in to small pieces then boiled at 100°C with 2% wt NaOH for 3 hours. The suspension was then filtered using vacuum filtration to remove all liquid. The resulting solid was mixed in blender to break down large fibre. Then, papers were boil again with 2% wt NaOH and 7% wt H_2O_2 for another 2 hours. These methods were performed in order to remove colors and impurities from the paper. Next, the sample was repeatedly washed using distilled water until neutral then dried in 60°C oven overnight.

2.3.2 Acid hydrolysis

5 g of cellulose was prepared in 50ML beaker with a few drops of distilled water. 85% cold H_3PO_4 was slowly added to the beaker and stirred using glass rod. H_3PO_4 was added and stirred until all piece of cellulose were completely dissolved. The suspension obtained was viscous and semi-transparent as shown in figure 2-1.

Figure 2-1 Acid hydrolysis of cellulose

2.3.3 Regeneration of amorphous cellulose

Different concentration of $Bi(NO_3)_3$ solution were prepared. 1 g, 2.5 g and 5 g of $Bi(NO_3)_3$ powder were dissolved in HNO_3, then were diluted with 500 ml distilled water. Then, 15000 rpm high speed homogenizer was used to homogenize prepared $Bi(NO_3)_3$ solution each with 5 g of amorphous cellulose (AC) (Figure 2-2). After getting $BiPO_4$/AC suspensions, acidity was attempted to be completely washed out from AC by repeatedly stirring the suspension, pouring out liquid and adding more distilled water until the solution was neutralized. In order for $BiPO_4$/AC to be dispersible in binding material, all liquid was removed by centrifugation until it has a white-paste texture as shown in Figure 2-3.

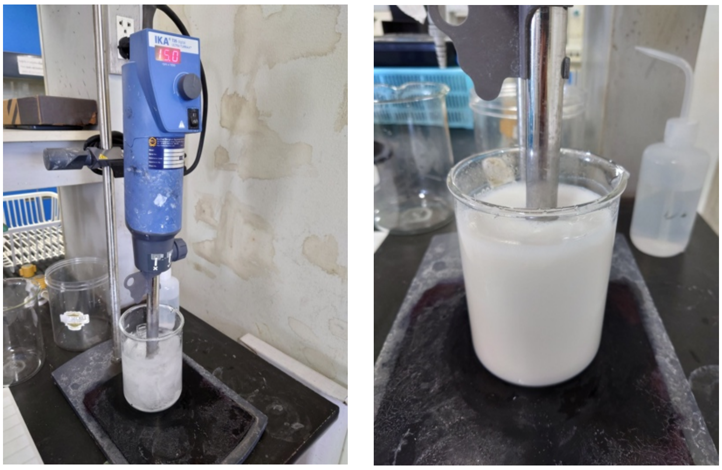
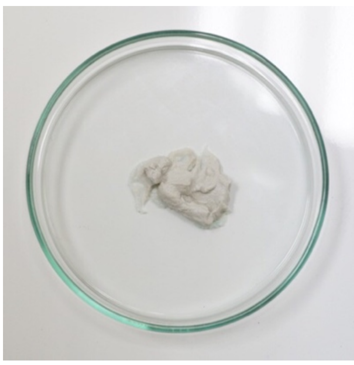


Figure 2-2 Regeneration of $BiPO_4$/AC from $Bi(NO_3)_3$ solution and amorphous cellulose from used envelop paper.


(a.)


(b.)


(c.)

Figure 2-3 $BiPO_4$/AC paste that were regenerate from the $Bi(NO_3)_3$ powder : amorphous cellulose at ratio (a.) 1:5 (b.) 1:2 (c.) 1:1

The %solid of each filler were obtained by weighting certain amount of $BiPO_4$/AC paste sample then the samples were dried in the oven until all solutions were removed. The weight of the dried $BiPO_4$/AC samples were measure and the %solid were calculated from:

$$\%solid\ filler = \frac{weight\ of\ dry\ BiPO_4 - AC}{weight\ of BiPO^4 - AC\ paste} \times 100 \quad (4)$$

2.3.4 Structure and chemical properties investigation

The chemical structure and properties of $BiPO_4$/AC were investigated by Scanning Electron Microscope with Energy Dispersive X-ray Fluorescence (SEM/EDX) and Energy-dispersive X-ray spectroscopy (EDS)

2.3.5 X-ray shielding ability test

2.3.5.1 Investigation of the silicone rubber and $BiPO_4$/AC ratio

Ratio of dry $BiPO_4$/amorphous cellulose filler from 2.3.3 and RTV(Room Temperature Vulcanizing) silicon rubber for coating mixture were investigated at 1:2, 1:4 and 1:7 respectively. The weight of filler paste were calculated from the formula;

$$filler = \frac{weight\ of\ SR\ \times\ formula\ ratio}{solid\ content\ factor} \quad (5)$$

Then the calculated amount of $BiPO_4$/AC filler and silicone rubber were mixed and stirred until the paste was well homogenized. After that, each mixture were coated on 10 × 10 cm 100% cotton specimen using plastic pad frame and stainless ruler (Figure 2-4)

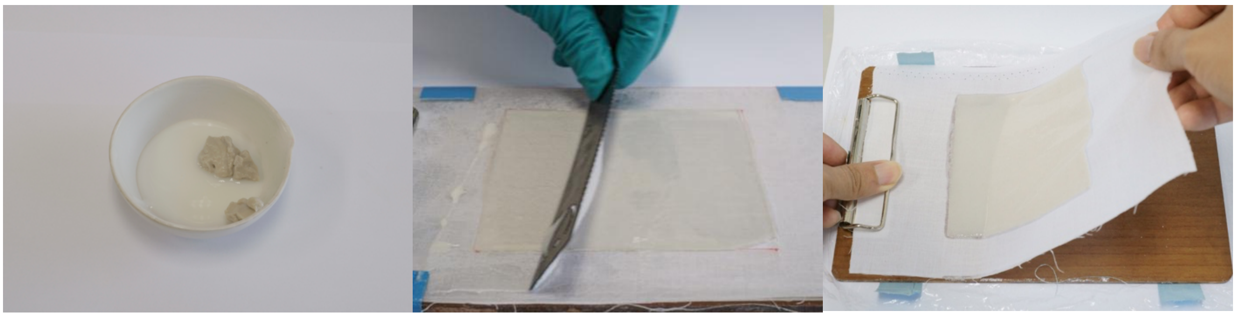


Figure 2-4 $BiPO_4$/AC-SR coating on 10 × 10 100% cotton fabric using clear plastic pad frame and stainless ruler

2.3.5.2 Fabric coating preparation

$BiPO_4$/AC-SR ratio 1:7 and 1:4 were chosen as the main proportion of the coating material as it was more homogeneous and shown a better coating texture compared with ratio 1:2. 8 samples were prepared from 3 different ratio of $BiPO_4$/AC filler with coating method referred from 2.3.5.1. Coating formulation are shown in Table 2-1. There are 2 control samples; one with 100% silicone rubber coated and one casting from $BiPO_4$ powder and silicone rubber without addition of amorphous cellulose.

Table 2-1 Coating formulation of 100% cotton fabric specimen for x-ray attenuation ability test

No.	BP-AC ratio	BP/AC-SR ratio	%solid filler (wt.%)	Silicon (g)	Filler (g)	Solid filler (g)
BP1	1 : 5	1 : 4	20.9	7.210	8.651	1.808
BP2	1 : 2	1 : 4	16.9	6.421	9.686	1.636
BP3	1 : 1	1 : 4	19.4	7.891	10.152	1.969
BP4	1 : 5	1 : 7	20.9	9.629	6.598	1.378
BP5	1 : 2	1 : 7	16.9	7.558	6.408	1.082
BP6	1 : 1	1 : 7	19.4	9.561	7.132	1.383
Control 1	no	no	0	11.523	0	0
Control 2	1 : 0	no	100	13.236	0.818	0.818

2.3.5.3 Sample test by X-ray generator

All samples were being test by MGC41 X-ray generator machine at 30, 40 and 60 kV and the exposure at 1.3 mA. The distance between the sample and the x-ray generator was set at 1 m. The samples were being test 3 times and the average results were used to calculate x-ray attenuation efficiency (μ) and %RAR using the equation (2) and (3).

Chapter 3

Result and discussion

3.2 Result from SEM and EDS

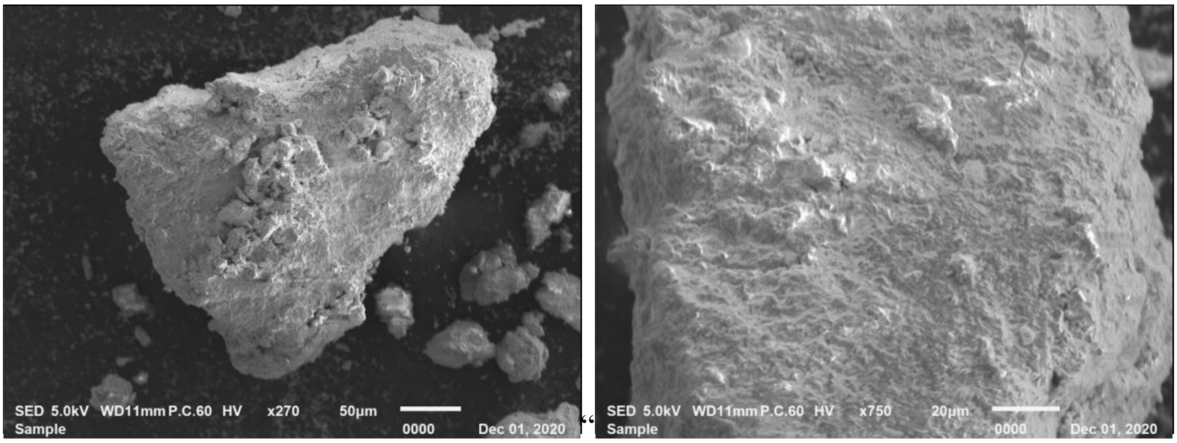


Figure 3-1 SEM of 1:5 $BiPO_4$-AC

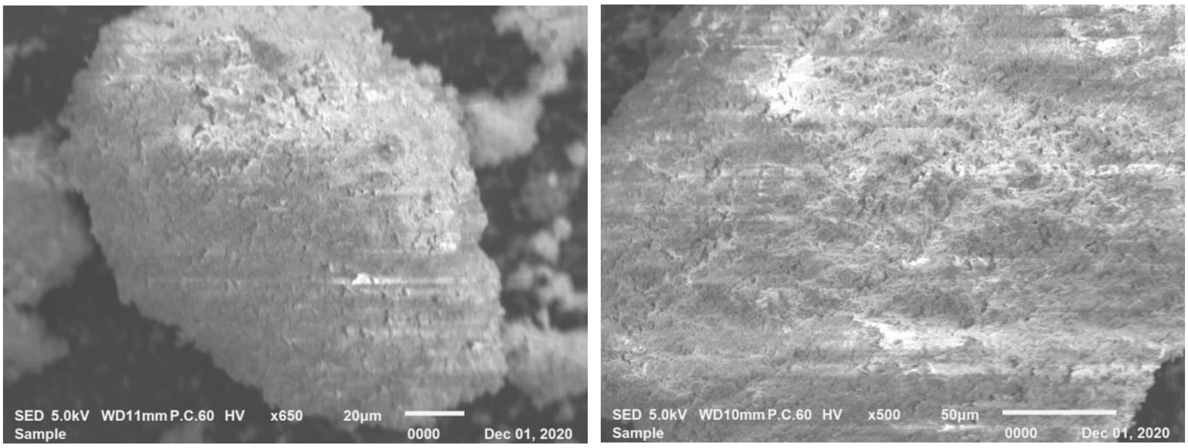


Figure 3-2 SEM of 1:2 $BiPO_4$-AC

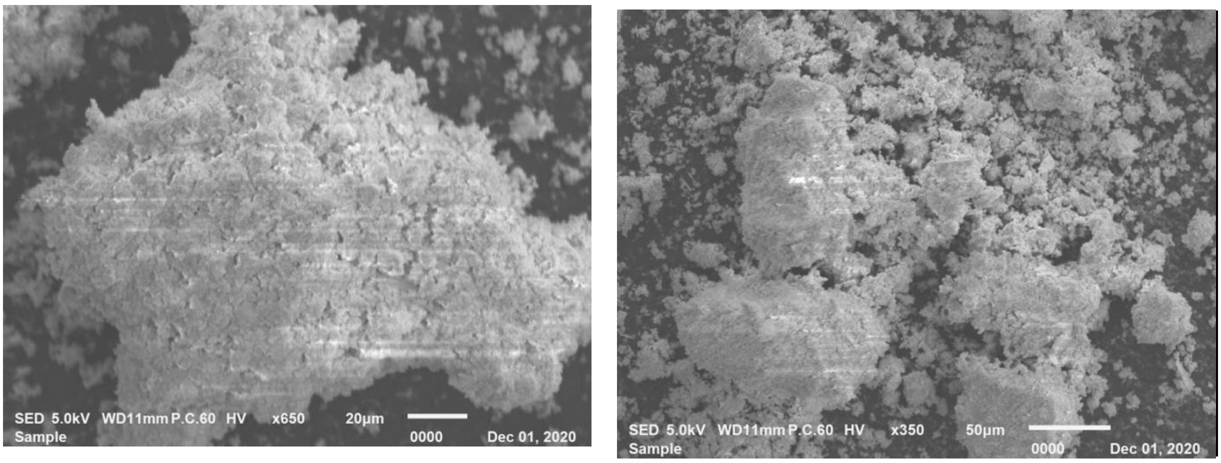


Figure 3-3 SEM of 1:1 $BiPO_4$-AC

Structural analysis from the Scanning Electron Microscope (SEM) of the different $BiPO_4$-AC in figure 3-1, figure 3-2 and figure 3-2 has shown that the sample with higher ratio of AC, 1:5, tend to have more homogeneous texture while sample with lower AC resulted in the breaking of the molecule into small pieces. This can be observed during the preparation step as well as when the sample were let dry in the oven, the sample with higher AC content were much harder to be grind into powder.

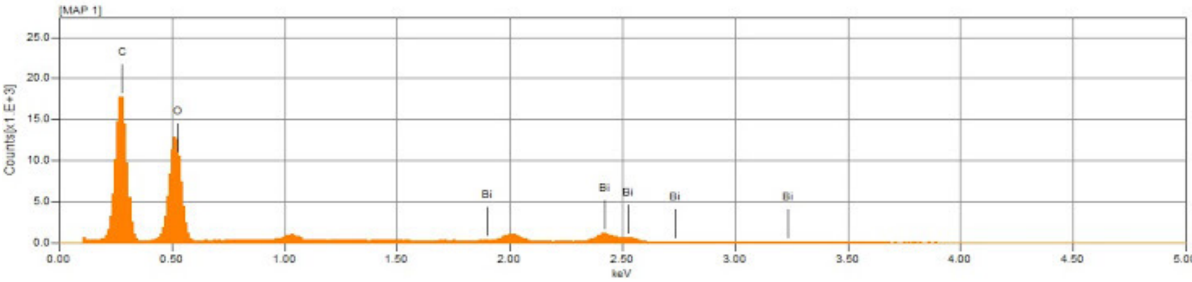


Figure 3-4 Element composition of 1:1 $BiPO_4$/AC analyzed by EDS

Table 3-1 Element composition of 1:1 $BiPO_4$/AC analyzed by EDS

Element	mass%	atom%
C	37.43	64.15
O	24.98	32.14
Bi*	37.59	3.70

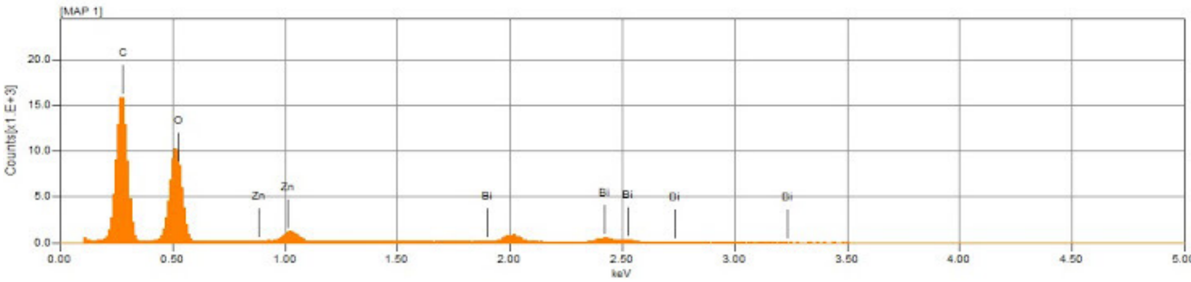


Figure 3-5 Element composition of 1:2 $BiPO_4$-AC analyzed by EDS

Table 3-1 Element composition of 1:1 $BiPO_4$/AC analyzed by EDS

Element	mass%	Atom%
C	45.97	67.17
O	26.83	29.43
Zn	6.05	1.63
Bi*	21.14	1.78

The result from energy-dispersive X-ray spectroscopy (EDS) of different ratio of 1:1 and 1:2 $BiPO_4$-AC (Figure 3-4 and 3-5) has shown that mass% and atom% of Bi were proportional to the ratio of Bi in the sample.

3.2 X-ray shielding efficiency of $BiPO_4$/AC-SR samples

Weight and thickness of all 6 samples were measured in order to calculate the content of $BiPO_4$ in each sample. Table 3-3 indicated that the sample with highest $BiPO_4$ content is BP3, followed by Control 2, BP6, BP2, BP1, BP5, BP4 and control 1 respectively. Theoretically, the attenuation properties of matters is proportional to the thickness and the atomic number Z of the matrices. Since all sample has similar thickness and contain the same kind of absorber metal Bi, the shielding ability is expected to be increased respective to the amount of $BiPO_4$ contain in the sample.

Table 3-3 Coating proportion including amount of $BiPO_4$ of 8 samples.

No.	BP-AC ratio	BP/AC-SR ratio	fabric mass	coated fabric mass	applied coated mass	BiPO4 in coating (g)	Thick. Avg (mm)
BP1	1 : 5	1 : 4	2.3938	13.5855	11.1917	0.213	0.943
BP2	1 : 2	1 : 4	2.2253	10.2604	8.0351	0.272	0.806
BP3	1 : 1	1 : 4	2.4975	12.9318	10.4343	0.569	1.026
BP4	1 : 5	1 : 7	2.1875	12.0805	9.8930	0.140	0.975
BP5	1 : 2	1 : 7	2.3542	10.1836	7.8294	0.202	1.005
BP6	1 : 1	1 : 7	2.3402	12.3155	9.9753	0.413	0.960
Control 1	no	no	2.3760	11.0948	8.7188	0.000	0.901
Control 2	1 : 0	no	2.3731	11.8722	9.4991	0.553	0.794

X-ray attenuation efficiency (μ) and radiation attenuation ratio (%RAR) of samples obtained from average of 3 measurement were stated in Table 3-4. The result shown the expected trend as the shielding ability of the sample decreased as the x-ray intensity increased. Furthermore, sample BP3 which contained highest amount of $BiPO_4$ (0.569 g) obtained the highest attenuation ratio, followed by BP6, Control 2, BP5, BP2, BP1, BP4 and control 1 respective (Figure 3-6 and Table 3-4)

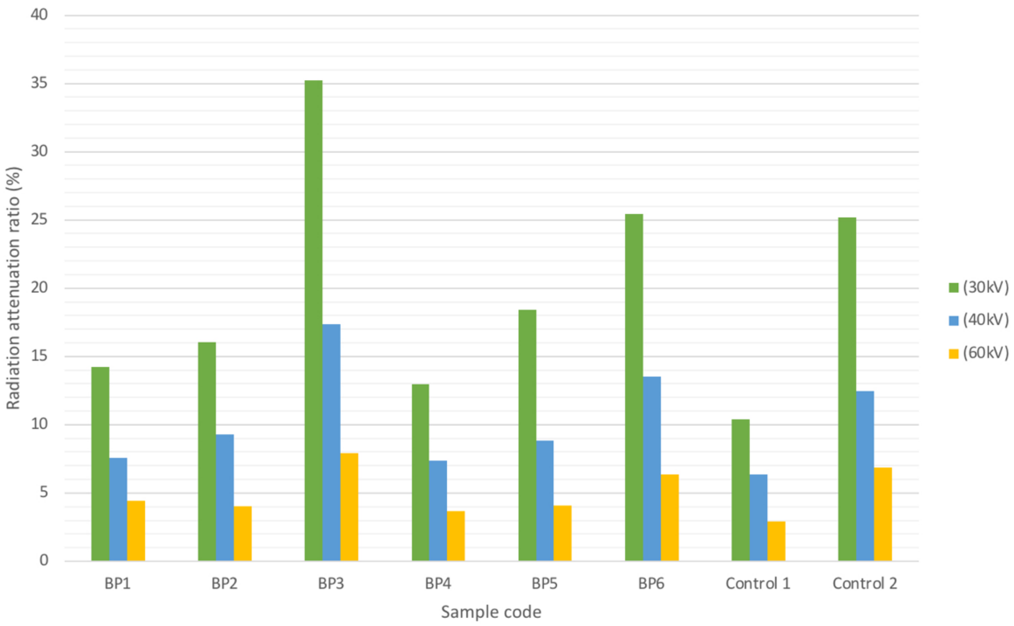


Figure 3-6 Graph representing comparison of radiation attenuation ratio (%RAR) of 6 samples

No.	BP-AC ratio	BP/AC-SR ratio	BiPO4 in coating (g)	%RAR (30kV)	%RAR (40kV)	%RAR (60kV)	u (30kV)	u (40kV)	u (60kV)
BP1	1 : 5	1 : 4	0.213	14.21	7.55	4.46	0.1625	0.0832	0.0484
BP2	1 : 2	1 : 4	0.272	16.06	9.29	4.06	0.2172	0.1209	0.0514
BP3	1 : 1	1 : 4	0.569	35.23	17.37	7.92	0.4234	0.1859	0.0804
BP4	1 : 5	1 : 7	0.140	12.98	7.38	3.69	0.1425	0.0786	0.0385
BP5	1 : 2	1 : 7	0.202	18.43	8.85	4.10	0.2027	0.0922	0.0416
BP6	1 : 1	1 : 7	0.413	25.46	13.52	6.36	0.3062	0.1514	0.0685
Control 1	no	no	0.000	10.39	6.34	2.92	0.1217	0.0727	0.0334
Control 2	1 : 0	no	0.553	25.19	12.48	6.87	0.3655	0.1679	0.0904

Table 3-4 X-ray attenuation efficiency (μ) and radiation attenuation ratio (%RAR) of 6 samples

Control 1, the sample that contains only silicone rubber has shown %RAR of 10.39% at 30kV, 6.34% at 40kV 2.92% at 60kV, which is the lowest out of all samples. This indicated that silicone rubber itself has very low ability to shield the radiation. Hence, the addition of $BiPO_4$/AC has enhanced the shielding performance. However, there were samples that did not follow the expected trend such as BP6 and BP5 that has shown better shielding performance comparing to the samples that had less $BiPO_4$ content.

Figure 3-7 shown 6 samples that were prepared from the coating formulation in Table 2-1. Theoretically, the homogeneous texture of the sample would be proportional to the ratio of the filler and silicone rubber, more cellulose filler would result in having more suspension in the surface. As you can see from Figure 3-7, samples with $BiPO_4$/AC-SR ratio of 1:4 (BP1, BP2 and BP3) have less homogeneous texture. However, an error could also occur during the mixing process. When the mixture of $BiPO_4$/AC-SR was not mixed well enough, Bi atom could not disperse evenly in the sample, resulted in an less effective attenuation ability. Therefore, the dispersion of absorber can affect the shielding ability of the material as well. Considering control 2, which contained only $BiPO_4$-SR resulted in less %RAR comparing to amorphous cellulose composited BP6 that contained lower $BiPO_4$, can indicated that amorphous cellulose took part in the absorber dispersion in matrices.

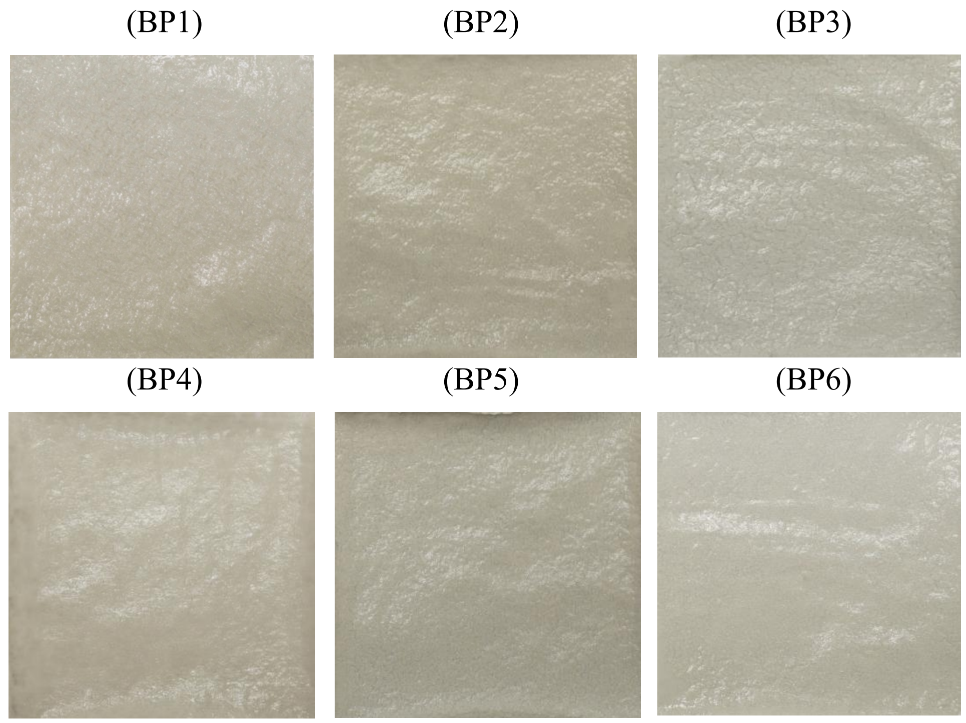


Figure 3-7 $BiPO_4$/AC-SR coated samples

Chapter 4
Conclusions

$BiPO_4$/Amorphous cellulose as an x-ray shielding material made with $Bi(NO_3)_3$ powder and amorphous cellulose from used brown envelope were investigated. Although the maximum %RAR obtained from the investigation at approximately 35% were comparatively low, but considering only 0.569 g of $BiPO_4$ present in the sample, this number can indicate an effective shielding efficiency. Furthermore, the result also showed predictable trend such that the shielding efficiency increased as content of $BiPO_4$ increased. Moreover, higher ratio of cellulose in $BiPO_4$-AC also resulted in more homogeneous texture of the substance, showing that amorphous cellulose has a good binding ability that could enhance the strength of the absorber-composited compound in other matrices as in this case, silicone rubber. Therefore, we can conclude that $BiPO_4$/AC solution can be develop further as an x-ray shielding absorber.

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