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Detection of X-rays by Polymer Composites of Lead Oxide with Silicone Rubber and Polystyrene

¹Suman, ²Sunita Dahiya, ³Kulvinder Singh, ⁴S.K. Chaudhary, ⁵Meena Devi

¹Research Scholar, ²Associate Professor, ³Associate Professor, ⁴Professor, ⁵Research Scholar

Department of Physics,

BM University, Asthal Bohar, Rohtak. India (124001)

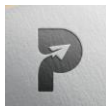
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Abstract

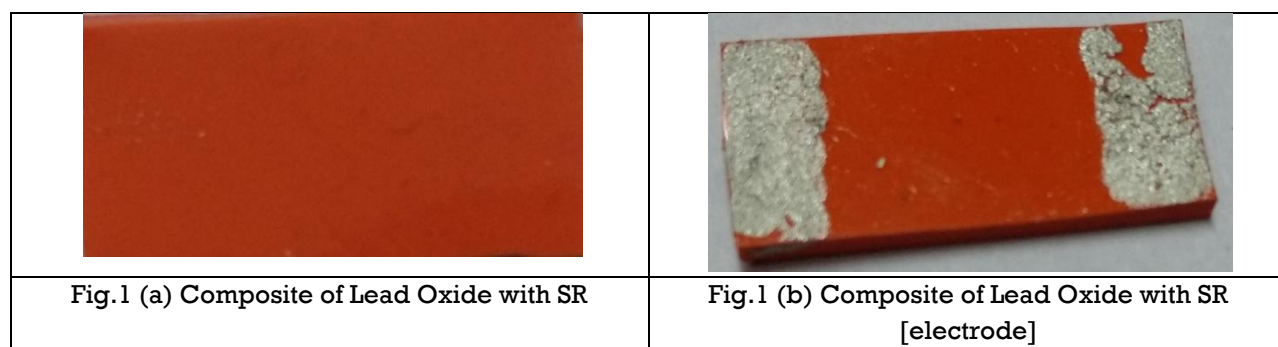
Polymer composites of lead oxide with silicone rubber (SR) and polystyrene (PS) were prepared and cut in the form of thin sheets. These sheets were subjected to X-ray switching studies under different values of electric field. Both the composites were found to have stable X-ray sensing and low rise and fall time making the material good for imaging applications. Mobility life time products of these composites were obtained by iterative method. It is found that these composites have high mobility life time product, low thermally generated charges at room temperature and high photoelectric absorption of X-rays in the range of 35 KeV and hence composites of lead oxide with these materials are proved to be suitable as well as good for high energy photons detection and imaging.

Keywords: Composites, Lead oxide, PS, SR, Switching, X-ray detector.

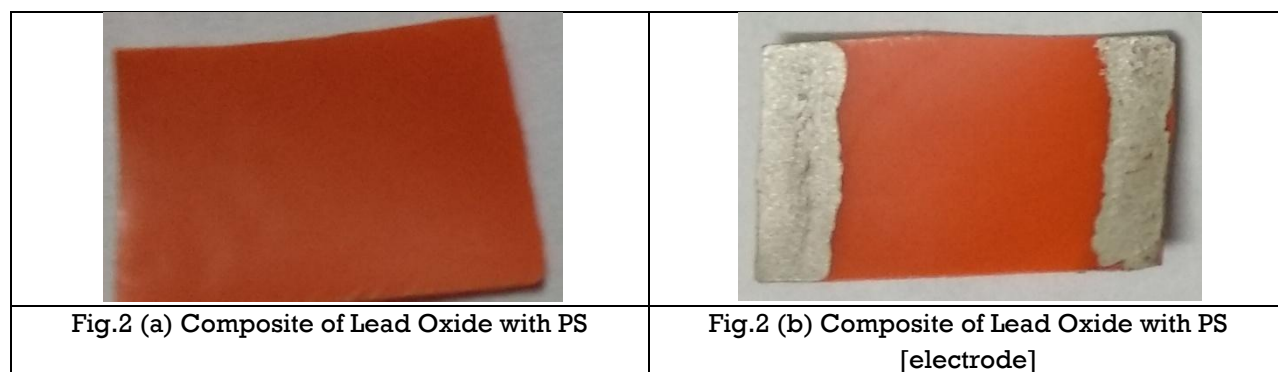
Introduction: X-rays have large number of applications which includes X-ray diffractions (XRD), X-ray microscopy, X-ray imaging etc. [1-4]. The important component of instruments used in these applications is X-ray detector. Large numbers of detectors are used to detect X-rays [5-6]. The selection of material for X-ray detector is based upon photoelectric conversion, mobility life time product, stability, band gap and atomic number. Lead oxide has high atomic number (82) which is useful for maximum absorption of X-ray energy. It also has high conversion efficiency for converting X-ray charge carriers. It is semi conducting oxide material having band gap between the range of 1.7 to 2.1 eV [7]. Hence lead oxide can be used for making solid state X-ray detector at room temperature. Due to increasing applications of detectors, another quality of the detector named as mechanical flexibility, is now also looked upon. For example, in dental radiography, if detector plate is flexible, it will be of great convenience for taking images of infected tooth. In connection to this application large number of polymer composites materials were investigated for increasing mechanical strength of the material used for detection of X-rays [8]. In present study we have fabricated the polymer composites of lead oxide with silicone rubber (SR) and polystyrene (PS). The SR and PS were selected due to their following properties like low density, low thermal expansion coefficient [9], good durability for weather changes and high electrical sensitivity which is good for keeping dark current low. The selection of SR was specifically giving flexibility to the detector.



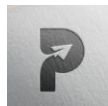
Experimental details: For preparation of composites of lead oxide with SR and PS, lead oxide (Pb_3O_4) was taken and fine grinded by using agate mortar and pestle. No additive was used during this process. Care was taken to keep the powder dry. SR was in liquid form so it was mixed with Pb_3O_4 by adding curing agent in it. SR (murtisil 1010 silicone rubber RTV) was mixed with 2.2 gm of curing agent (1010). Fine grinded powder of lead oxide was then added to this mixture. This uniform mixture was kept for 48 to 72 hrs to settle down. The samples were cut in the form of thin sheets and were made electrodes by using silver paste. These sheets are flexible just like rubber as shown in fig.1(a) and (b) respectively.



No hot pressing was done to prevent any mechanical damage to the sheets. For making samples of PS, sheets of PS were first broken and changed into granules. Granules of PS were dissolved in chloroform as it is easily soluble in chloroform. Slurry formed was mixed with fine powder of Pb_3O_4 . The mixture obtained was left to settle down for a week. Hard composite sheets of Pb_3O_4 with PS were obtained and made electrodes by using silver paste as shown in fig 2.(a) and (b) respectively.



Three samples of composites of lead oxide with SR and PS were prepared by using different concentrations of SR and PS such as 60%, 80 % and 90%. For measuring the response time of the detector at room temperature the samples were subjected to X-ray switching studies. X-ray source with copper target was used. X-ray generator was operated at 30 KV having 10 mA plate current. To chop X-ray beam, switching rotor device was used. X-rays were blocked by 4mm semi circular lead disc. Stepper motor was used to control rotation (micro processor P89C51RD2). Keithley



6485picometer was used for measuring photo current[10]. Switching curves of PS and SR are shown in fig 3 (a) and (b) respectively.

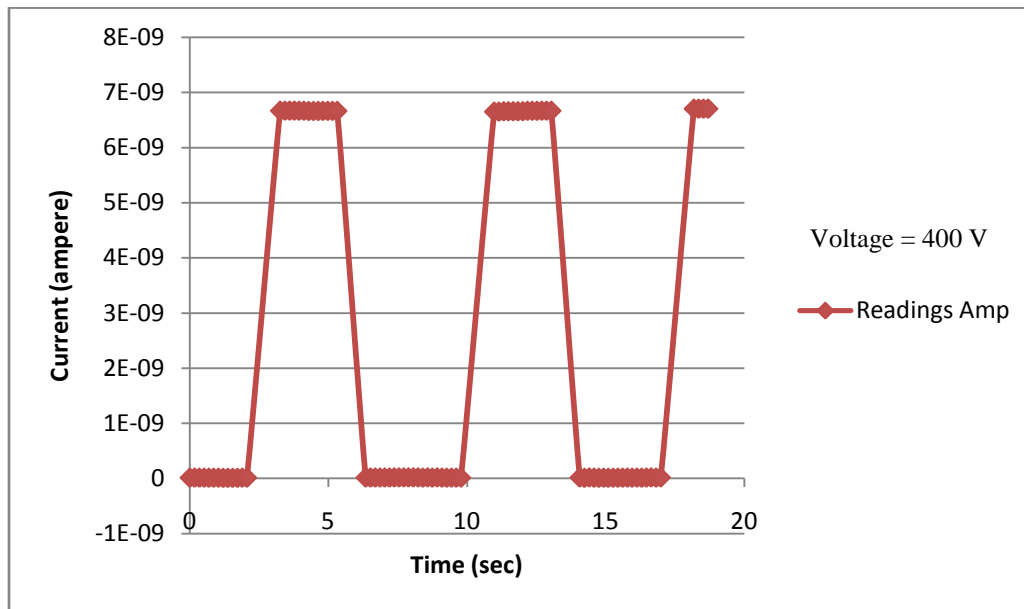


Fig 3(a) Switching curves of composite of lead oxide with PS

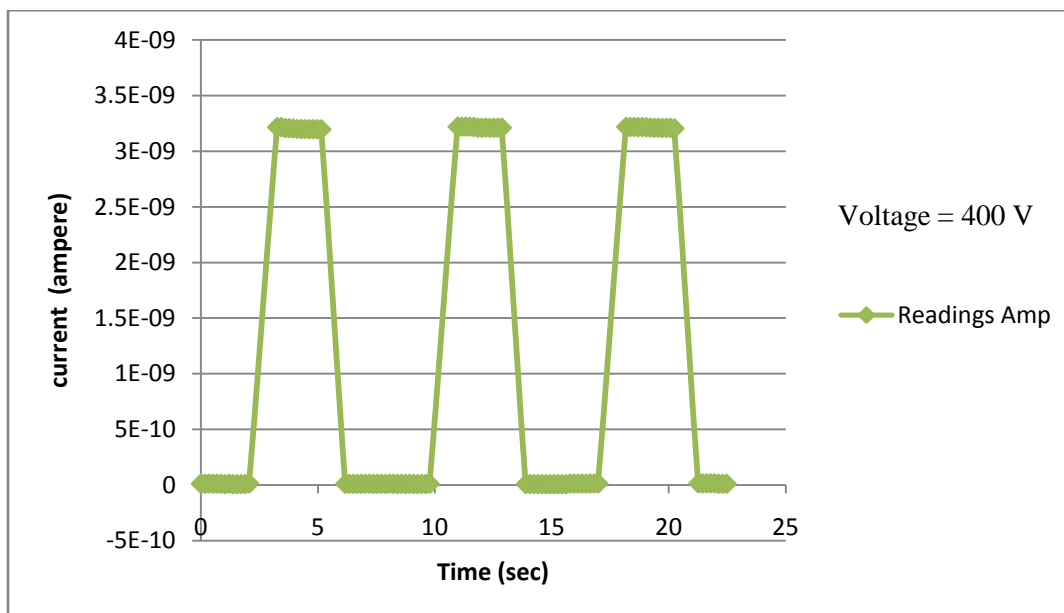


Fig.3(b) Switching curves of composite of lead oxide with SR



Scanning electron microscopy (SEM) is a powerful technique in the examination of materials. We can obtain high magnification images with a good depth of field and can also analyze individual crystals. The magnification of the image in this technique is the ratio of the size of the screen to the size of the area scanned on the specimen [11]. Scanning electron micrographs were taken on fractured portion of these sheets as shown in Fig.4

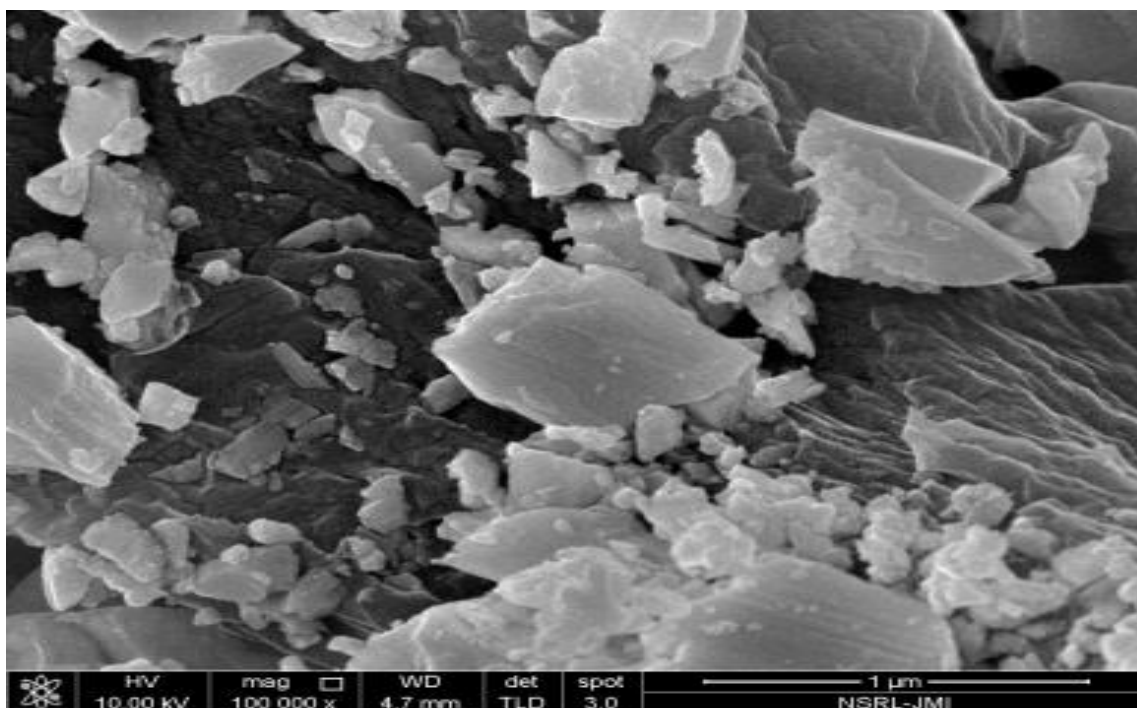


Fig.4 SEM micrograph

Energy dispersive X-ray spectroscopy (EDS) is an analytical technique used for the chemical analysis or chemical characterization of a sample [12]. The samples in the form of thin sheets were subjected to energy dispersion spectroscopy to confirm the chemical composition of these sheets as shown in Fig 5.

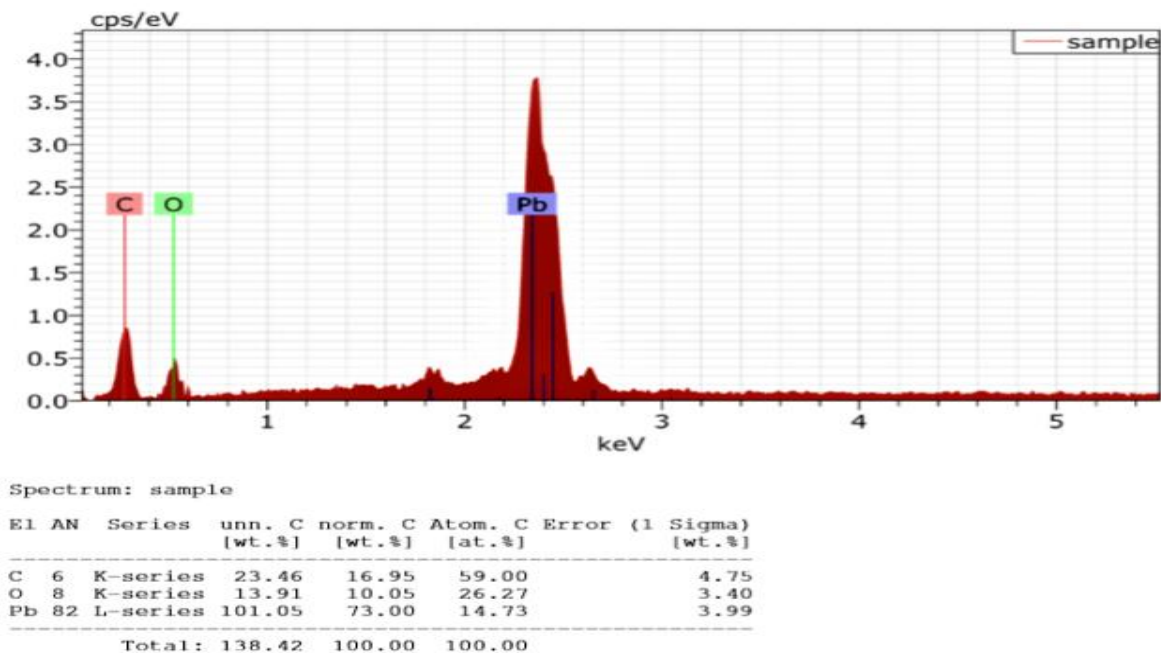


Fig.5 EDS spectrum and chemical composition

Particle size of composites of lead oxide with SR and PS were determined using XRD technique. Their respective XRD peaks and Gaussian fit are as shown in Fig. 6 (a) and 6 (b)

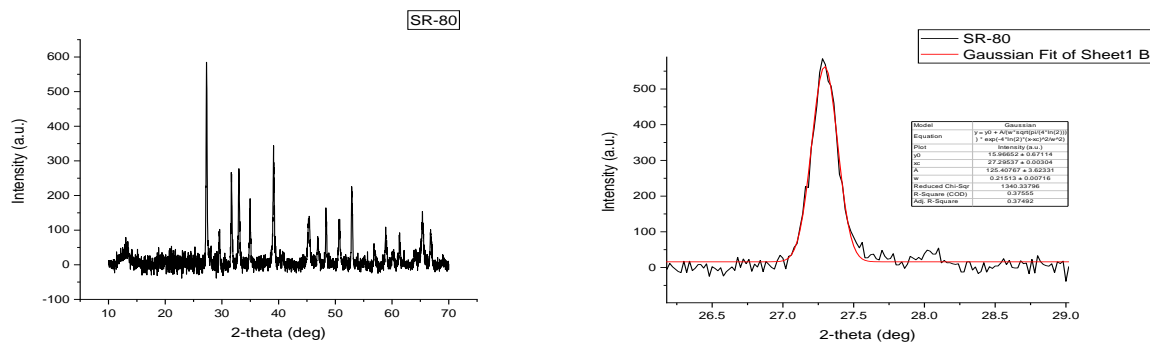


Fig .6(a) XRD peaks and Gaussian fit of Pb_3O_4 with SR

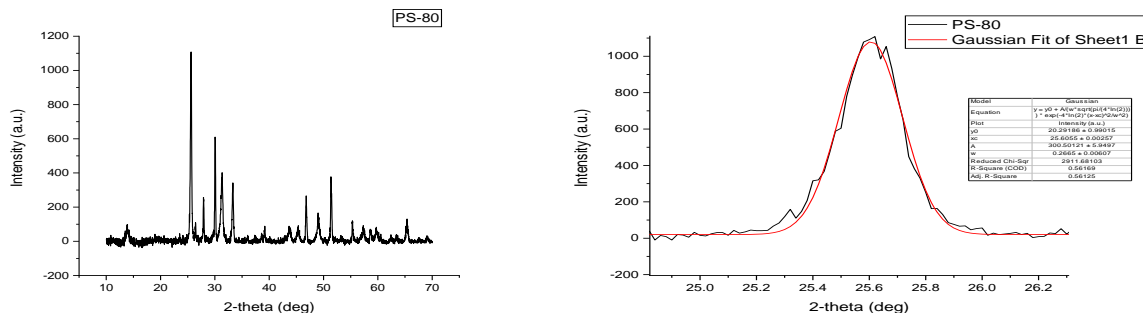
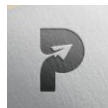


Fig .6(b) XRD peaks and Gaussian fit of Pb_3O_4 with PS

Crystallite size was determined by using Paul Scherrer equation [13]. The calculated average values of particle size for pure lead oxide and its composites with SR and PS are shown in table 1.

Table 1. Particle size

| Material | Particle Size (in nanometer) |
|-------------------|------------------------------|
| Pure lead oxide | 28.45nm |
| Pb_3O_4 with SR | 29 nm |
| Pb_3O_4 with PS | 23.56 nm |

Result and discussion: The most important parameter of detector material is its mobility life time product. This parameter was calculated by using modified hecht equation [14-16]

$$Q = Q_o \left(\frac{\mu\tau E}{d} \right) [1 - \exp(-d / \mu E)] + kE$$

In this equation, Q represents charge collection by photo generation, μ is mobility of charge carriers, τ is life time, E is electric field, d is thickness of sample, k is correction coefficient. The term kE is added for the charge collection by the base material for sample holding. In our case base materials are SR and PS. The value of correction coefficient 'k' will depend on intensity of the radiation reaching at the base material and charge generation by the medium due to highly ionizing radiation. Mobility life time product ($\mu\tau$) for both the composites of lead oxide with SR and PS were determined by iterative method.

In this method large numbers of iterations were done by changing values of $\mu\tau$ and k, up to the values where the normalized error became minimum and stable. The experimental results approached to theoretical results.



Experimental graphs were compared with theoretical graphs obtained by iterative method and were found to be nearly same. The results (graphs) of both the composites PS and SR are shown in Fig. 7 (a) & 7 (b) respectively.

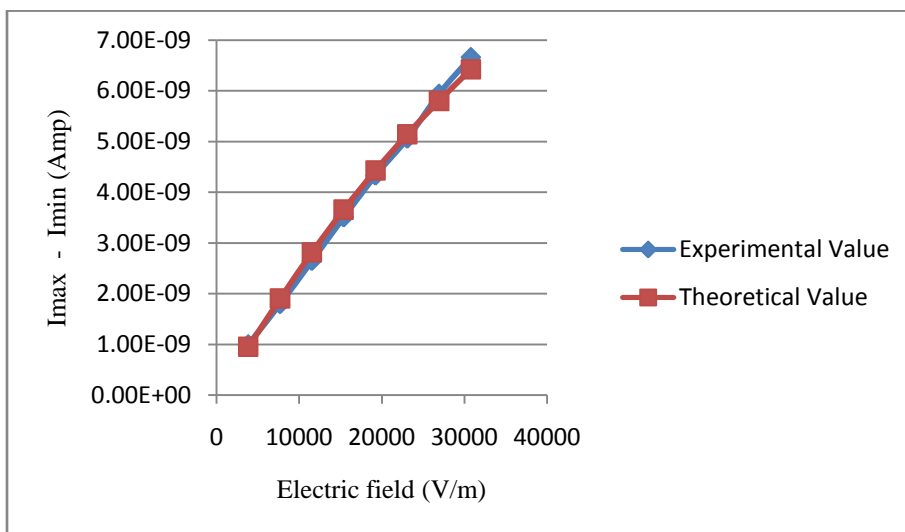


fig. 7 (a) Experimental and theoretical fitting of Photocurrent with electric field in PS composite.

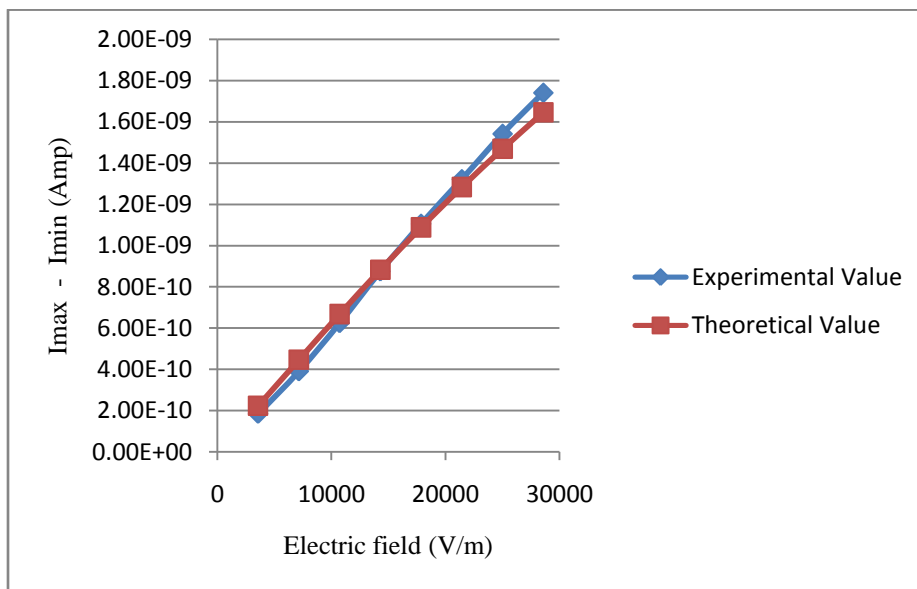


fig. 7 (b) Experimental and theoretical fitting of Photocurrent with electric field in SR composite

Electrical conduction in these composites is facilitated by hopping process. The average values of mobility life time product for both the composites of lead oxide with SR and PS were calculated using iterative method and are listed in Table 2.



Table 2. Mobility life time product of composites of lead oxide with SR and PS.

| Composite | Mobility life time product |
|--------------------|--|
| Lead Oxide with SR | $29 \times 10^{-8} \text{ m}^2/\text{V}$ |
| Lead Oxide with PS | $29.3 \times 10^{-8} \text{ m}^2/\text{V}$ |

Mobility life time product of the composites of lead oxides with SR was found to be smaller than its composites with PS. Hence it is revealed that the response time of the detector made by composites of lead oxide with PS is more as compared to its composites with SR. But SR has other advantageous characteristic that is its flexibility and food grade. A detector made of by using SR polymer can be easily used while taking dental X-ray without any harmful effect. In case of sample preparation of SR, care must be taken during adding the quantity of curing agent. If the curing agent is added in less quantity, it leaves some strands unlocked and if it is added in more quantity, it stays in between interstitial space and causes charge trapping which leads to low $\mu\tau$ factor. The curing agent contains isopropanol. This isopropanol changes the molecular structure of lead oxide. Due to this curing efficiency decreases, which effects the X-ray detection efficiency.

Conclusion: The experimental studies revealed that the composites of lead oxide with PS show better X-ray sensitivity as compared to its composites with SR. Rise and fall time of PS composites are also better then SR composites. X-ray detection efficiency of PS composites was also found more than the composites of SR. hence composites of lead oxide with PS can be used for high energy photons detection and imaging.

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