ANKARA UNIVERSITY
INSTITUTE OF NUCLEAR SCIENCES

MASTER'S THESIS

DEVELOPMENT OF NOVEL SHIELDING MATERIAL BASED ON EPOXY MATRIX LOADED BY HIGH-Z CHEMICAL POWDERS USED FOR MAMMOGRAPHIC, DIAGNOSTIC AND INDUSTRIAL X AND GAMMA RAY ENERGY RANGE, AND DETERMINATION OF ITS PHOTON ATTENUATION PROPERTIES BY IONIZATION CHAMBER METHOD WITH USE OF STANDARD BEAM QUALITIES

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THESIS APPROVAL

The thesis entitled "Development of Novel Shielding Material Based on Epoxy Matrix Loaded by High-Z Chemical Powders Used for Mammographic, Diagnostic and Industrial X and Gamma Ray Energy Range, and Determination of Its Photon Attenuation Properties by Ionization Chamber Method with Use of Standard Beam Qualities" which prepared by İbrahim DEMİREL in partial fulfilment of the requirements for the degree of Master of Science in Health Physics Program of the Medical Physics Department of Institute of Nuclear Sciences in Ankara University and has been examined and approved by the undersigned jury members.

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I hereby declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, required by these rules and conduct, I have fully cited and referenced all material and results that are not original to this work.

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ABSTRACT

Master's Thesis

DEVELOPMENT OF NOVEL SHIELDING MATERIAL BASED ON EPOXY MATRIX LOADED BY HIGH-Z CHEMICAL POWDERS USED FOR MAMMOGRAPHIC, DIAGNOSTIC AND INDUSTRIAL X AND GAMMA RAY ENERGY RANGE, AND DETERMINATION OF ITS PHOTON ATTENUATION PROPERTIES BY IONIZATION CHAMBER METHOD WITH USE OF STANDARD BEAM QUALITIES

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The shielding against ionizing radiation is an important issue in the medical and industrial radiation applications as a passive safety element. In recent years, with fast development of material science new composite materials have been developed and they are increasingly used in this field. Those materials are competitively employed together with classical radiation shielding materials such as lead and iron metals, and even they are also replaced by metals due to some practical reasons. In this thesis, it was aimed to produce a novel composite shielding material based on epoxy matrix by adding lead oxide powder. The suitability of its use in mammographic, radiological and industrial x and gamma ray energy ranges was tested by ionization chamber method. The new shielding samples were produced by mixing lead oxide powder and a suitable epoxy matrix, thus resulting in at least 99% homogeneity in which the homogeneity of samples was determined using the radiological images taken by a-Si/CsI based digital imaging detector. In the production procedure, the samples were produced using different loading ratios of lead oxide/epoxy, different shapes and thicknesses. Physical densities of the samples were determined by using a special balance kit. The attenuation properties of the produced samples such as lead equivalence, attenuation coefficient and HVL values were measured on standard narrow beam qualities according to IEC 61331-1 protocol, using the experimental setups installed in the radiometric benches. It was observed that the 20 % PbO loaded sample with 7mm thickness showed no transmission and shielded all incoming photons in mammography energy range (20-30 kVp), the 60% PbO loaded sample with 4mm thickness is found to be equivalent to 0.5mm lead metal for radiology (80-100 kVp). To test the samples industrial energy range, an ISO s-137Cs (0.662 MeV) source was used to determine lead equivalence, attenuation coefficient, and HVLs. Then the mechanical properties of the samples were determined and the results obtained for impact strength are satisfactorily to demonstrate their durability. All results obtained for the samples indicated that, this new shielding material can be used in some medical and industrial applications.

2019, 56 Pages

Key Words: Shielding material, radiation protection, lead equivalence, nuclear technology, lead oxide

ÖZET

Yüksek Lisans Tezi

MAMOGRAFİK, TANISAL VE ENDÜSTRİYEL X- VE GAMA ENERJİ ARALIKLARINDA KULLANIM İÇİN EPOKSİ MATRİSE YÜKSEK Z'Lİ KİMYASAL TOZ KATKILANARAK YENİ BİR ZIRH MALZEMESİ GELİŞTİRİLMESİ VE FOTON ZAYIFLATMA ÖZELLİKLERİNİN STANDART DEMET KALİTELRİNDE İYON ODASI YÖNTEMİYLE BELİRLENMESİ

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İyonlaştırıcı radyasyona karşı zırhlama malzemesi kullanımı, pasif bir güvenlik elemanı olarak tıbbi ve endüstriyel radyasyon uygulamalarında önemli bir yer teşkil eder. Son yıllarda malzeme biliminin hızlı gelişimi ile birlikte bu alanda yeni kompozit malzemeler kullanılmaya başlanmıştır. Bu malzemeler, kurşun ve demir metalleri gibi klasik radyasyon zırhlayıcı malzemelerle birlikte rekabetçi bir şekilde ve hatta bazı pratik nedenlerden dolayı metaller yerine de kullanılırlar. Bu tezde, kurşun oksit tozuna ve epoksi matrisine dayalı yeni bir kompozit zırhlayıcı malzeme üretilmesi amaçlanmıştır. Mamografik, radyolojik ve endüstriyel enerji aralıklarındaki x ve gama ışınlarına karşı kullanımının uygunluğu, radyometrik tezgâhlarda iyon odası yöntemiyle test edilmiştir. Koruyucu numunelerin üretimi kurşun oksit tozunun uygun bir epoksi malzeme ile karıştırılma ve kalıplanma tekniğine dayanmaktadır, numunelerin homojenliği a-Si / CsI dijital görüntüleme detektörü kullanılarak en az % 99 homojenlik olacak şekilde elde edilmiştir. Numuneler farklı kurşun oksit / epoksi yükleme oranları, farklı şekiller ve kalınlıklar kullanılarak üretilmiştir. Örneklerin fiziksel yoğunlukları özel bir terazi seti kullanılarak belirlenmiştir. Üretilen numunelerin kurşun eşdeğerliği, zayıflama katsayısı ve HVL değerleri gibi foton zayıflama özellikleri, IEC 61331-1 protokolüne göre standart dar demet kalitelerinde tanımlanmıştır. 7 mm kalınlıkta % 20 PbO yüklü numunenin mamografi enerji aralığında (20-30 kVp) hiçbir doz değeri göstermediği ve gelen tüm fotonları durdurduğu, 4 mm kalınlıkta % 60 PbO yüklü numunenin radyoloji enerji aralığı (80-100 kVp) için 0.5 mm saf kurşun levhaya eşit olduğu gözlenmiştir. Örnekleri endüstriyel enerji aralığında test etmek amacıyla, kurşun eşdeğeri, zayıflama katsayısı ve HVL'lerini belirlemek için bir ISO s-137Cs (0.662 MeV) kaynağı kullanılmıştır. Ayrıca numunelerin darbelere dayanıklılığı için yapılan testlerde tatmin edici sonuçlara ulaşılmıştır. Numuneler için elde edilen tüm sonuçlar, bu yeni koruyucu malzemenin bazı tıbbi ve endüstriyel uygulamalarda kullanılabileceğini göstermiştir.

2019, 56 sayfa

Anahtar Kelimeler: Zırh malzemesi, radyasyondan korunma, kurşun eşdeğeri, nükleer teknoloji, kurşun oksit

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CONTENT

LIST OF SYMBOLS AND ABBREVIATIONS

Gy	**'Gray' Absorbed dose unit**
HVL	**Half Value Layer**
ICRP	**International Commission on Radiological Protection**
ICRU	**International Commission on Radiation Units**
IEC	**International Electrotechnical Commission**
ISO	**International Standards Organization**
kVp	**kilo voltage peak**
μ	**Linear attenuation coefficient**
μ/ρ	**Mass attenuation coefficient**
ρ	**Density**
δ_{Pb}	**Lead equivalent**
PbO	**Lead oxide**
RQR	**Radiation Qualities in Radiation beams emerging from the x-ray source assembly**

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1. INTRODUCTION

Radiation exposure situations are explained in three sections which are planned, emergency and existing exposures; planned situations are categorized in three groups; medical, occupational and public exposures and these are limited with dose limits (ICRP 103 Report, 2007). For obeying dose limits for planned exposures, it is necessary to optimize exposure time, exposure distance and shielding. Radiation protection with a shielding material is a passive safety mechanism against ionizing photon radiation. Lead, iron and steel are conventionally used materials for radiation protection. However, in recent years, with development of material science there are alternative shielding materials in the literature including polyurethane (Ni et.al, 2016), epoxy resin (Mkhaiber & Dheyaa, 2018), lead-free materials and lead composite materials (Eder et. al, 2010). Production of these innovative materials are very significant for development of nuclear shielding technologies but accurate testing of such shielding materials against the ionizing radiation, especially for photon radiation is also more important in terms of radiological safety. Although, in some studies, point gamma sources are used with scintillation or semiconductor detectors (Li et.al, 2017) in order to determine shielding effectiveness of the material. However, this type of measurement technique is not realistic when the point source is used because such transmission experiments do not meet the requirements of international standards and protocols (such as AAPM TG 61 and IEC 61331-1). This simple laboratory measurement technique with use of point sources does not guarantee the universality and reliability of the results obtained for the samples. Instead, it is a must to use the sources and x-ray systems comply in with ISO and IEC standards. Because the international standards in this subject recommend the use of a radiation beam equal to or greater than the material to be tested (Büermann 2016). The reason for this recommendation is to consider possible impurities and scattering effects within the material.

This master thesis was conducted in two main steps. In the first step, a novel shielding material based on two component epoxy matrix and lead oxide powder was produced in laboratory scale by using simple laboratory mixing techniques. In the production technique, we focused on the molded samples which can be formed in the desired shapes and thicknesses without any air bubbles, but with higher homogeneity.

The second step is to test the prepared shielding materials in mammographic, radiological and industrial energy ranges, respectively. To achieve this, the reference metallic materials with 99.9 % pure aluminum sheets and lead sheets were firstly used. Three different radiation sources in different energy ranges were used to test the shielding properties of the samples prepared in the first step. These energy ranges are, respectively, mammographic (20-30 kVp), radiological (40-100 kVp) and industrial (ISO s-^{137}Cs-661.6 keV). In addition, a digital imaging system operating in the radiological energy range was used to test the homogeneity and reproducibility of the prepared shielding materials. According to standard protocol, the radiation attenuation properties of the present composite shielding material were determined in terms of aluminum equivalent in the mammographic energy range, in terms of lead equivalent (δ_{Pb}) in radiological and industrial energy ranges. Before performing such measurements, the x-ray systems were tested in terms of kVp accuracy tests, 1st and 2nd HVL values for the beam qualities were determined. In order to obtain the radiation beam qualities in IEC and ISO protocols which will be explained in detail in the following sections of this study, additional filters are added to system, or kVp is normalized. In addition, many details such as measurement geometry, the required irradiation area, and optimum tube current are considered in accordance with the standards. In the measurement of dose and dose rates ion chambers with respect to their energy dependency were used. As a final goal; aluminum equivalence of samples in mammographic energy range, the HVL values and lead equivalence (δ_{Pb}) of the samples were determined by using transmission curves in both radiological RQR beam qualities defined in IEC protocol and industrial ISO-s ^{137}Cs(0.662MeV) source energy ranges.

There are five chapters in this thesis. After this Introduction chapter, the theoretical concepts and principles and the necessary basic theory the thesis are explained in the second chapter. In the third chapter, the material and method used are explained. In the fourth chapter, the results are explained with tables and graphs. In the last chapter, the obtained results are discussed and concluded.

2. THEORY

2.1 General Definitions

2.1.1 Absorbed dose

The total amount of energy that absorbed by the unit mass could be identified as an absorbed dose or only dose. The classical unit of the absorbed dose "rad" and it is deposition of 100 erg energy in a 1-gram media. In the international system of units (SI) absorbed dose unit is "gray (Gy)" and it is the deposition of 1 Joule energy in a 1-kilogram media (ICRP 103 publication 2007).

1 Gy= 1 J/kg= 100 rads

2.1.2 Dose equivalent or equivalent dose

The term dose equivalent is used for explaining radiation damage difference with respect to same amount of deposited energy. Radiation damage in an organism is not directly proportional to deposited energy namely absorbed doses, it also related to LET (linear energy transfer) value. This means, in the equal amounts of absorbed dose value, the radiation damage would be higher in the high-LET radiation values. Alpha particles and neutrons have high-LET values while photons and betas have low-LET values. This term calculated by multiplication of absorbed dose (D) with quality factor (Q) and donated by H. The classical unit of the dose equivalent is "rem".

H (dose equivalent) = D (absorbed dose) x Q (quality factor)

In the international system of units (SI) dose equivalent is "Sievert (Sv)" (ICRP 103 publication 2007).

Equivalent dose

The equivalent dose, H_T, to an organ or tissue, T, is defined in ICRP 60 and ICRU 51. For a single type of radiation, R, it is the product of a radiation weighting factor, w_R, for radiation R and the organ dose, D_T, thus:

H_T (equivalent dose) = D_T (absorbed dose) x w_R (radiation weighting factor)

Unit: J/kg. The special name for the unit of equivalent dose is Sievert (Sv). The radiation weighting factor, w_R, allows for differences in the relative biological effectiveness of the incident radiation in producing stochastic effects at low doses in tissue or organ, T. For x- ray energies used in diagnostic radiology, w_R is taken to be unity.

There is a small difference between dose equivalent and equivalent dose as a concept. In fact, there is not seen the difference for photon radiations but it can be effective for other type radiations such as charged particles and neutrons because there might be unnegligible difference between Q and w_R depending on the type and its energy.

2.1.3 Exposure

This term is valid for x-ray and gamma ray interactions with air, and it is used for identification of the amount of ionization that produced. This quantity is measurable directly with collection of the electrical charges, while it is not possible for other mediums such as human body. The classical unit of exposure is called "roentgen (R)", and it is not unit of absorbed dose, but it is convertible to dose with a suitable adjustment. 1 roentgen equals to 2.58×10^{-4} coulomb charge in 1 kg of dry air due to x and gamma rays. In the international system of units (SI) the unit of exposure is called "X", and it is defined as generation of 1 coulomb charge in the 1 kg of dry air. 1 exposure (X) is equals to 3876 roentgen(R) (ICRP 103 publication 2007). The exposure can be related with the dose quantity, e.g 1 Roentgent equals to 8.76 mGy

2.1.4 Kerma

Kerma is kinetic energy released in material or energy loss in the material or media. While uncharged particles or waves such as neutrons or photons transverse in a medium they transfer their kinetic energy to charged particles in a unit mass. Unit of the kerma is J/kg. For photons, it can be accepted that dose value accepted equal to kerma value (ICRP 103 publication 2007).

2.1.5 Half value layer (HVL)

First HVL or just HVL is the thickness of the material which reduces the dose rate to half of its initial value (ICRU Report 74 2005).

Second half value layer (2nd HVL)

Second HVL is obtained by subtraction of the first HVL from the thickness of the material that reduces the dose rate to quarter of its initial value (ICRU Report 74 2005).

Homogeneity factor (h)

Homogeneity factor is the algebraic ratio of first HVL to second HVL (ICRU Report 74 2005).

2.1.6 Primary and secondary radiation

Primary radiation is incoming x or gamma radiation from its source without any scattering, interaction or change in direction. Secondary radiation is incoming x or gamma radiation that scattered or interacted from a material such as human body, phantom or any other scattering mediums.

2.1.7 Radiation attenuator plates and filtration

Attenuator plates such as iron or lead or other alloys are x or gamma ray absorber materials that placed between the beam and the detector for reducing the intensity of the beam.

The x-ray spectrum which consists of bremsstrahlung radiation and characteristic x-rays is partially filtered by the materials made of the x-ray tube. This filtration is called inherent filtration. Besides to inherent filtration, the added filter to increase the average energy of the x-ray spectrum and filter the low-energy photons is called additional filtration. The summation of inherent filtration and additional filtration gives total filtration. In order to determine inherent filtration of x-ray tube according to TS-8661-1 ISO-4037-1 standard, firstly, HVL value need to be determined at 60 kVp without using any additional filtration. Then by using obtained HVL value inherent filtration could be achieved in the following table. Determination of filtration is an important point for determining the characteristics of x ray system and high voltage generator. For instance, 1% change in x-ray tube voltage can lead to altering output of the filtered beam, say 15 % (TS-8661-2 ISO 4037-2 2004).

Table 2.1 Relationship between HVL and inherent filtration. (TS-8661-1 ISO 4037-1 2004)

1st HVL Values at 60 kVp (mm-Al)	**Inherent Filtration (mm-Al)**
0.33	0.25
0.38	0.3
0.54	0.4
0.67	0.5
0.82	0.6
1.02	0.8
1.15	1.0
1.54	1.5
1.83	2
2.11	2.5
2.35	3
2.56	3.5
2.75	4
2,94	4.5
3.08	5
3.35	6
3.56	7

2.1.8 Peak kilo voltage of x-ray tube system

Peak kilo voltage (kVp) is the applied voltage to x-ray tube. The value of applied voltage gives the maximum energy, with increasing kVp the maximum energy and the average energy of x-ray spectrum increases. However, the increasing kVp does not affect the place of characteristic x-ray on the spectrum, it only alters peak height.

2.1.9 Physical density

Density is mass per unit volume in a sample. Density is denoted by symbol "ρ" or "D". Algebraic value of density of a substance could be found by division of mass "m" to volume "V".

2.2 Photon Sources

2.2.1 Monochromatic photon sources

Photons with discrete energy spectrum are called monochromatic photon sources. The best example of monochromatic radiation sources are radioactive sources such as gamma sources**.** In this thesis study, ^{137}Cs (0.662 MeV) monochromatic photon source according to ISO 4037-1 was used for irradiations.

2.2.2 Polychromatic photon sources

Photon sources with continuous energy spectrum are called polychromatic photon sources. X-rays are examples of polychromatic photon sources. During this thesis, x-rays were used as a polychromatic photon source in radiological and mammographic energy intervals. In this thesis, the x-ray system with target/filter Mo/Mo and Mo/Rh was used in mammographic range and the medical diagnostic x-ray system with tungsten anode according to IEC 61267 were used in the irradiations.

2.2.3 Production of x-rays in mammography and diagnostic equipment

X-rays can be produced in different purposes such as industrial or medical with use of different kVp ranges in this thesis, the principle for mammographic and diagnostic x-ray equipment is briefly described but the subject is not limited with this.

X-rays are produced in x-ray tube which is consists of two main parts positive anode and cathode applied by potentials. The cathode part consists of filament wire with high resistance. When voltage is applied to the filament, thermionic electrons are released. These electrons are accelerated by the electric field from the cathode to the anode. The anode is selected from materials with high Z and high melting points, such as tungsten (W), molybdenum (Mo) or rhodium (Rh). In general anode has a rotational motion to dissipate heat quickly. X-ray tube is vacuumed in order to reduce interactions of electrons with air molecules. When negative electrons are reached to the target atom at kept positive voltage, the electron interacts with force field of the nucleus. These interactions such as acceleration of electron or deflection of the electron result in production of x-rays. In some cases, incoming electron interacts with bound electron which results in characteristic x-ray production.

Characteristic x-rays

It is the emission of x-ray due to electron transitions between atomic orbits. When an energetic free electron hits to electron of the target material, bound electron in the inner shell is removed from its orbit and a vacancy occurs. Because of this vacancy in the inner shell, the atom becomes unstable. For re-stability of the atom a bound electron from outer shell fills the vacancy. While this transition x-ray emission is released equal to difference between binding energies of two levels. It is called as characteristic since binding energies of an atom is unique to itself. This uniqueness results in discrete x-ray spectrum depending on the anode material (Bushberg 2012).

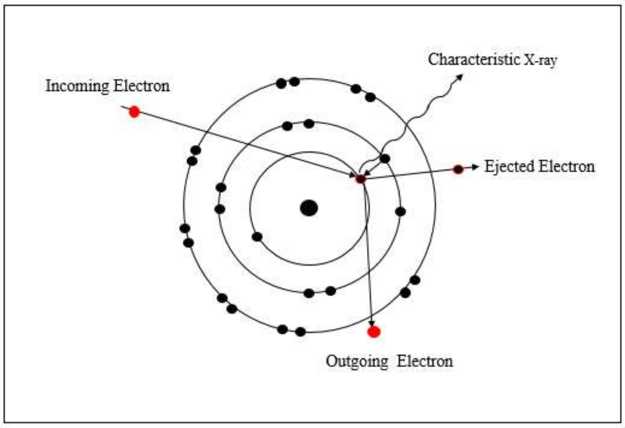

Figure 2.1 Characteristic x-ray emission, the electron emits energy while filling vacancy in the lower shell

Bremsstrahlung radiation

When negative electrons are reached to the target atom, the electron interacts with force field of the nucleus. These interactions such as deceleration of electron or deflection of the electron result in production of x-rays. During the close interaction of electron and nucleus, electron is lost some of its energy; lost energy is converted to bremsstrahlung radiation. This braking effect (Bremsstrahlung) on electron could be different in size with respect to strength of the interaction, thus resulting in a continuous the spectrum (Bushberg 2012).

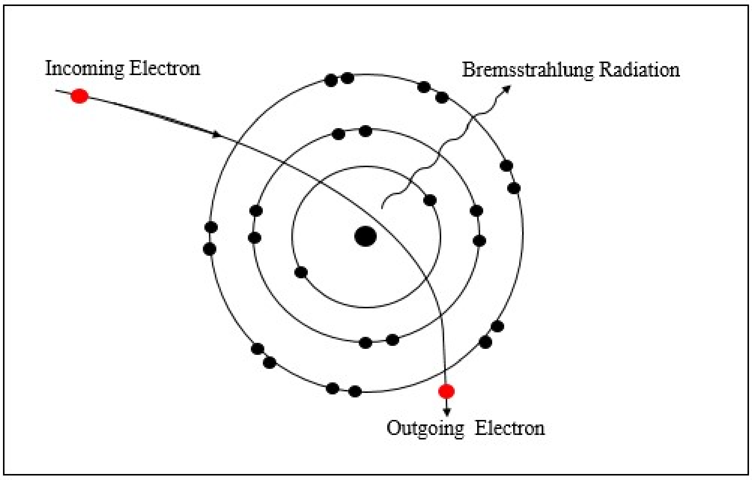

Figure 2.2 Production of x-rays by accelerated electron emits bremsstrahlung

Auger electrons

In some cases, characteristic x-rays cannot escape from the atom, and interact with weakly bound electrons in outer shells of the atom. As a result of this interaction, electron ejects from the atom this electron is named Auger electron and this situation called Auger effect.

2.3 Radiation Interactions with Matter

2.3.1 Compton scattering

Compton Effect or Compton scattering is an important phenomenon that explains interaction of electromagnetic wave with a particle. When a gamma ray or x-ray interacts with a weakly bound orbital electron, the electron is scattered and incoming electromagnetic beam loses some of its energy so the wavelength of the wave increases while its energy decreases. In Figure 2.3 schematic diagram for Compton scattering is shown, where λ_i initial wave length of the incoming electromagnetic wave and λ_f final wavelength (Martin 2013).

$$\sigma_{comp} \approx \text{constant} Z / A \tag{2.1}$$

The probability of Compton Scattering is related to Z/A ratio target material.

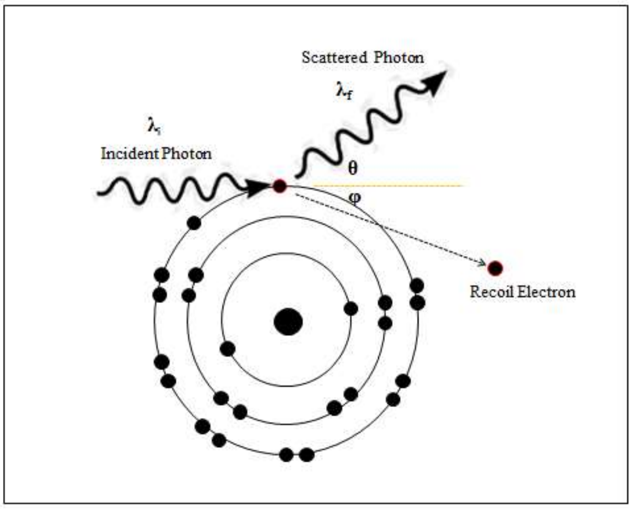

Figure 2.3 Compton scattering schematic diagram

2.3.2 Photoelectric effect

When an electromagnetic photon beam such as x and gamma ray interacts with a tightly bound electron of an atom, electron absorbs nearly all energy of the photon and released from the atom. This electron is called photoelectron and this situation is called photoelectric effect. Although photoelectric effect could be seen in different energy ranges of the electromagnetic spectrum for example visible, infrared, visible, ultraviolet; nuclear physics is interested in energetic photons like x-rays and gamma rays (Martin 2013).

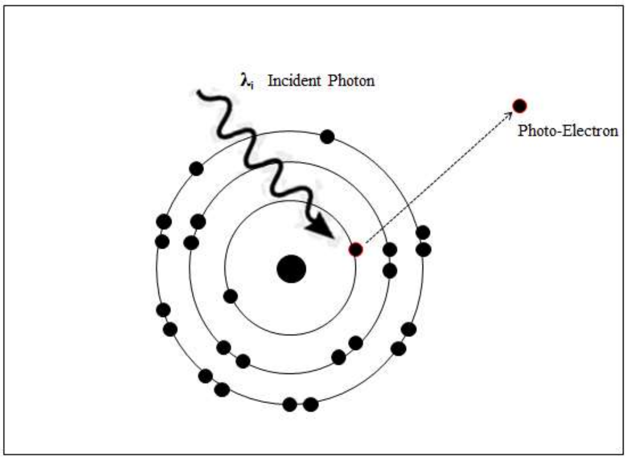

Figure 2.4 Photoelectric effect schematic diagram

$$\sigma_{ph} \approx \text{constant} \cdot \frac{Z^{4.5}}{E^{3.5}} \tag{2.2}$$

The probability of photoelectric effect is related to Z of the target material and incoming photon energy.

$$\mu_{m} = \frac{\mu}{\rho}\left(\sigma_{ph} + \sigma_{Comp} + \sigma_{pp}\right) \times \frac{N_{Av}}{A} \tag{2.3}$$

Mass attenuation coefficient is proportional to probability of photoelectric effect, Compton scattering and pair production and atomic mass of the material.

2.3.3 Photon transmission

While a photon beam such as gamma ray or x-ray passes through an absorber material, its intensity is reduced by the material. This absorption or attenuation can occur with Compton scattering, photoelectric effect and pair production. These physical interaction processes are sometimes very competitive but the Compton scatterings are expected in all energies. However, the photoelectric effect is dominantly in low energies of 100-200 keV depending material density and its effective atomic number. The pair production interaction occurs in nuclear field if the photon energy is greater than 1022 keV. As result, the decrease in photon intensity is also dependent on the material properties such as its density, thickness and atomic number (Turner 2010). The algebraic equation for intensity is represented by Beer-Lambert Law as follows by taking into account photon buildup effect of the material at a given energy;

$$I = B \cdot I_0 e^{-\mu x} \tag{2.4}$$

In equation above, I_0 is the intensity of incoming photon beam, I is the intensity of photon beam that passed from the attenuator, μ is the linear attenuation coefficient for the attenuator material and x is the thickness of the attenuator material. B (μx, E) is the build factor, which can be determined empirically or Monte Carlo simulation at a given energy for a material. μx represents mean free path for photons that transmit through that material. The terms absorption and attenuation are generally used interchangeably but there is a small difference between them. The term attenuation includes both scattering and absorption. If we organize equation (2.4) and taking logarithm, then equation takes the following form;

$$\ln\left(\frac{I}{I_0} \cdot \frac{1}{B}\right) = -\mu x \tag{2.5}$$

As previously described, HVL is the thickness of the material which reduces the dose rate to half of its initial value. In ideal case (i.e., B=1), If I_0 is chosen as half of the I and put into equation (2.5) takes the form as follows;

$$\ln(2) = \mu x_{1/2} \tag{2.6}$$

Reformulating equation (2.6);

$$x_{1/2} = \frac{\mu}{\ln(2)} \tag{2.7}$$

As seen in equation (2.7), both half value layer and linear attenuation coefficient are depending on energy of photons. In addition to HVL and linear attenuation coefficient, mass attenuation coefficient is another term which is independent of density and phase of the matter. For example, in broad beam conditions, B factor cannot be neglected for especially more scattering media for photons such as water.

In this thesis study, HVL, attenuation coefficient and density of the prepared samples are calculated for determination of photon attenuation parameters in different energy ranges.

2.4 Shielding of Photon Sources

2.4.1 Standard method for radiation shielding

TS EN61331-1:2004 European norm or international version IEC-61331-1:1994 both explain protective devices against diagnostic medical x-ray radiation and determination of attenuation properties of materials. The attenuation properties are given in terms of:

- Attenuation ratio;

- Build-up factor;

- Lead equivalence

These three parameters could be achieved by applying overall procedure in both the narrow beam and broad beam conditions. Alternative to broad beam, inverse broad beam conditions which explained by Büermann (Büermann 2009) could also applicable. The difficulty of broad beam is that the shielding materials need to be minimum 50cm x 50cm area. In the case of inverse broad beam conditions, a special farmer type ionization chamber is needed. In this thesis, only narrow beam geometry was applied and lead equivalence of the samples were obtained according the standard because it is produced small pieces of (10cm x 10cm) to save the consumed materials.

2.4.1.1 Narrow beam conditions and geometry

For narrow beam condition the test object needs to 10 cm x 10 cm dimensions and the irradiation area should be 2 cm out of the edges of the object. Moreover, the detector diameter has to less than 5cm. The standard geometry for narrow beam is shown in the Figure 2.5. K_e is air kerma rate in the narrow beam which is used for determination of lead equivalence. For this purpose, reference lead sheet materials are used to obtain transmission curve, then it’s compared with the test materials. The determination of lead equivalences is done in different x-ray beam energies.

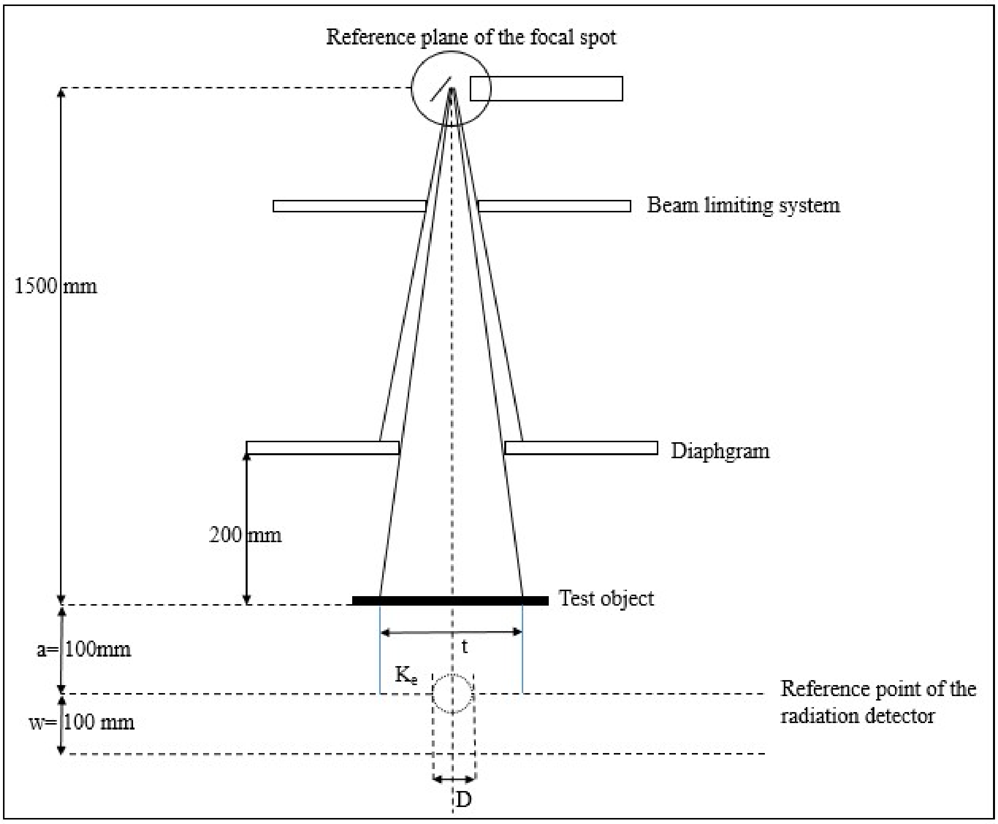

Figure 2.5 Narrow beam geometry

2.4.1.2 Broad beam conditions and geometry

For broad beam condition the test object needs to 50 cm x 50 cm dimensions and the irradiation area should be larger than the sample. Moreover, the detector diameter has to less than 5cm. The standard geometry for broad beam is shown in the Figure 2.6. Where;

K_i is air kerma rate with attenuated broad beam.

K_0 is air kerma rate without any attenuation.

K_c is air kerma rate in the center of the broad beam between the sample and focal point.

K_{oc} is air kerma rate outside the broad beam in the same distance to focal point as K_c.

K_s is air kerma rate inside the broad beam, but after the radiation beam which limited by diaphragm.

K_{1s} is air kerma rate is attenuated broad beam by the sample which is in the same distance from focal point with K_s.

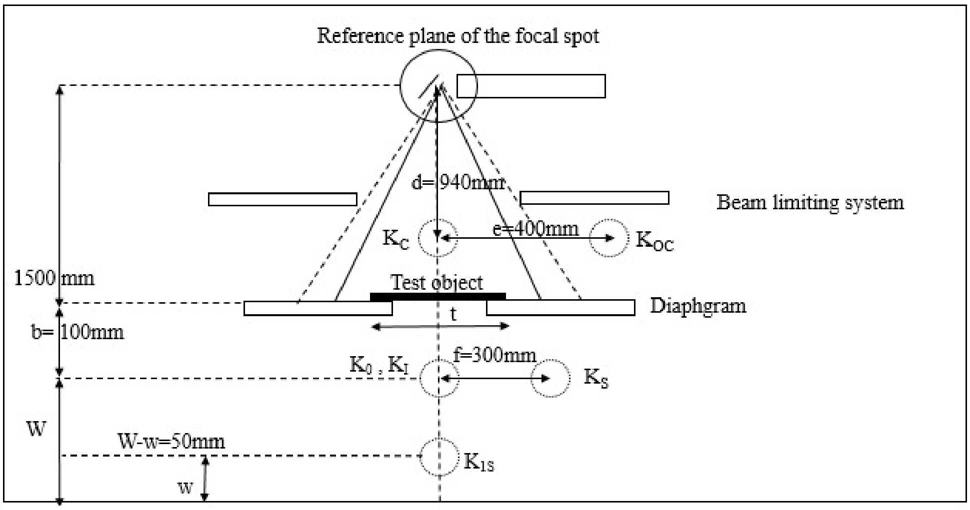

Figure 2.6 Broad beam geometry

2.5 Some International Beam Qualities Used in the Thesis

2.5.1 Standardized beam qualities in IEC-61331-1 for x-ray protective materials

X-ray beam qualities are identified by their total filtration with respect to material such as copper or aluminum in millimeters and first HVL. According to IEC 61331-1 standard it's advised (Yücel et al. 2016) to use beam energies 80 and 100 kVp energies for radiological practices.

Table 2.2 Standard beam qualities

X-ray tube voltage (kVp)	Total filtration* (mm Cu)	First HVL (mm Al) nominal
30	0.05	0.99
50	0.05	1.81
80	0.15	2.77
100	0.25	3.44
150	0.7	5.17
200	1.2	14.16
250	1.8	16.8
300	2.5	18.6
400	3.5	20.8

*These are also equal to 2.5 mm Al(total filtration) for beam qualities up to 150kVp.

2.5.2 Beam qualities explained in IEC-61267 protocol

In the IEC (International Electrotechnical Commission) 61267 protocol, standard radiation conditions are identified by the parameters such as anode material, anode angle, applied peak kilo voltage, total filtration, HVL, homogeneity factor and measurement distance. IEC 61267 protocol includes many standards with respect to system or system filtration material. Some of the standards and some of their properties are as follows;

Table 2.3 Some of radiation qualities explained in IEC- 61267 protocol

Radiation Quality	Radiation Description	Additional Filter Material	Application System
RQR	Radiation Qualities in Radiation beams emerging from the x-ray source assembly	Without Phantom	General radiography, fluoroscopy and dental applications
RQA	Radiation Qualities based on a Phantom	Aluminum Filter (Phantom)	General radiography, fluoroscopy and dental applications
RQT	Radiation Beam with an Added Filter	Copper Filter	Computed Tomography applications
RQR-M	Radiation Qualities in Radiation beams emerging from the x-ray source assembly	Without Phantom	Mammography applications
RQA-M	Radiation Qualities based on a Phantom	Aluminum Filter (Phantom)	Mammography applications

In this thesis study, RQR radiation qualities between RQR2 to RQR8 according to IEC 61267 were obtained in the presently used calibration system installed in a radiometric bench and these radiation qualities are used in the measurements. The properties of

RQR series in radiology energy range is given in the table below. RQR9 and RQR10 were not obtained intentionally for not to force high voltage generator.

Table 2.4 Specifications of RQR beam qualities (IEC 61267:2005)

Beam Quality	**Peak Kilo Voltage**	**1st HVL**	**2nd HVL**	**Homogeneity Factor (HF)**
RQR2	40	1.42	1.75	0.81
RQR3	50	1.78	2.34	0.76
RQR4	60	2.19	2.95	0.74
RQR5	70	2.58	3.63	0.71
RQR6	80	3.01	4.36	0.69
RQR7	90	3.48	5.12	0.68
RQR8	100	3.97	5.83	0.68
RQR9	120	5.00	7.35	0.68
RQR10	150	6.57	9.12	0.72

For providing standard RQR series, peak kilo voltage value has to be in the limits of ± 1.5 kV, homogeneity factor has to be in the limits of ± 0.03, 1st and 2nd HVL values have to be same as in Table 2.4. In addition, anode material has to be tungsten and anode angle has to be 12º. The distance between focal point to detector has to be equal or greater than 55 cm. The distance between detectors to additional filters has to be at least 5 times the diameter of the detector (IEC 61267 2005).

2.5.3 Properties of ^{137}Cs gamma-ray

Since the source should be as small as possible, it is mandatory to use radioactive material with sufficient activity per unit mass. In addition to activity per unit mass, the type of the radionuclide is also important for both use for calibrating the dose meters and testing the shielding efficiency of the materials. Collimated and capsulated ^{137}Cs was used in this thesis as a gamma ray source in order to determine lead equivalences of the samples. According to ISO 4037-3 standard, the measurement conditions need to be made between 1 to 3-meter distance in order to obtain adequate irradiation area. The following table represents radionuclides and their properties which are advised by the standard.

Table 2.5 Standard gamma ray sources and their properties (TS-8661-1 ISO 4037-1: 2004)

Type of Radionuclide	Energy of Radionuclide (keV)	Half-Life (days)	Specific Activity $Bqkg^{-1}$	Chemical Compound
^{60}**Co**	1173.2 1332.5	1925.5	$3.7x10^{15}$	Metal
^{137}**Cs**	661.6	11050	$8.51x10^{14}$	Chlorinated compound
^{241}**Am**	59.54	157788	$1.11x10^{14}$	Oxide

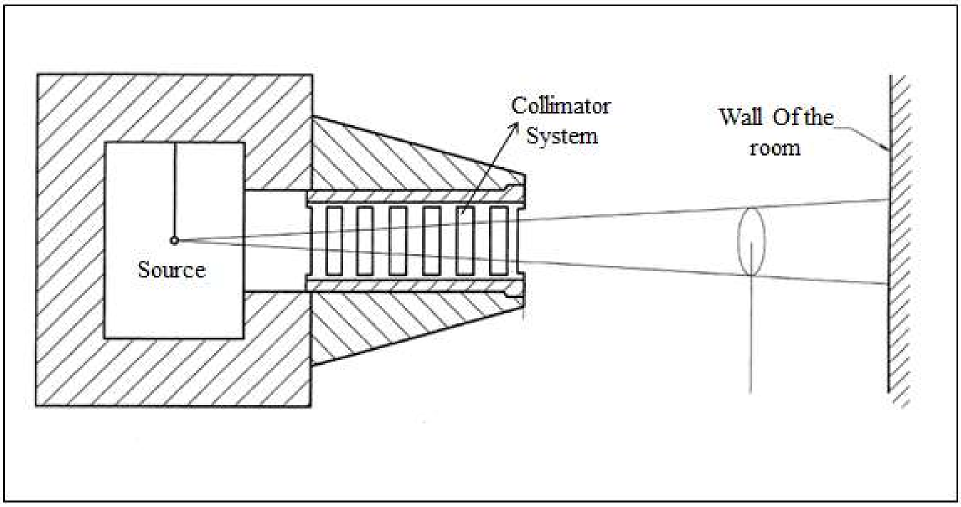

Figure 2.7 Collimated geometry for gamma sources (TS-8661-1 ISO 4037-1 2004)

2.6 Detectors Used in This Study

2.6.1 Ionization chambers

Ionization chambers are most common gas-filled detectors, beside GM counters and proportional counters. Ion chambers operate by evaluating the amount of ionization produced by the radiation while it goes through the active gas medium of the detector. Generally, these type of active detectors consists of two electrodes, an impermeable gas medium, external power supply connected to electrodes and a counter. When an ionizing radiation enters to active volume of the detector it gives its energy to gas atoms, so creates electrons and ions. After ionization process with effect of the electrical field electrons and ions start to move across to electrodes. With help of the suitable electronic system; current due to electron movement is measured (current mode) or charges are converted to pulses, (pulse mode) then pulses are counted. In both of the modes, ion chambers do not make energy differentiation; it only gives information about the presence of ionizing radiation. The classification of ion chambers could alter with respect to design of the electrodes such as parallel plate, cylindrical or spherical. Different designs and active detector volume could be used for different purposes. For example, for measuring low dose rates larger detector volumes are preferable or vice versa. In this thesis different brand and model ion chambers are used for determination of HVL, attenuation coefficient and lead equivalence of the samples.

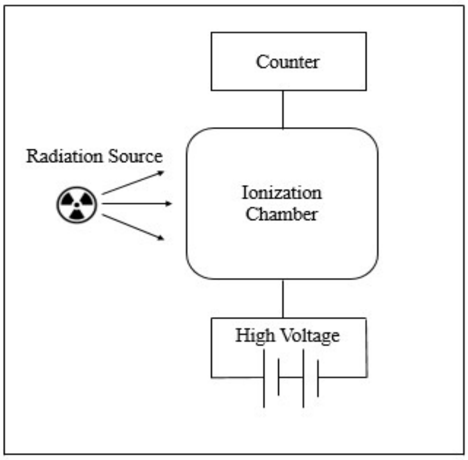

Figure 2.8 Schematic diagrams for ionization chambers with a counter

2.6.2 Scintillation detectors

Scintillation materials could be in the form of gases, liquids or solids. These forms could be organic and inorganic substances. When ionizing radiation interacts with scintillation material, energy of radiation converted to scintillation light in the visible or ultraviolet light photons. This scintillation light is converted to very few numbers of photoelectrons with low energies of 1-2 eV, so the multiplication is needed (Knoll 2012). For this purpose, photomultiplier tube which has dynodes with increasing voltage differences is used. Thus, proportional to intensity of the incoming radiation, amplified photoelectrons are produced. These photoelectrons create a signal pulse with respect to its energy; finally, pulses are saved in a spectrum with help of the multichannel analyzer. A scintillation detector was used for the thesis study, against the probability of gamma emitters in the lead oxide compound. This scintillation detector contains a NaI(Tl) crystal which is very common inorganic scintillator for gamma rays. NaI(Tl) crystal has both advantages and disadvantages. Advantages are high atomic number, high density ($3.67x10^3$ kg/m^3), reproducibility of desired dimensions, high efficiency. Disadvantages are sensitivity to temperature changes, hygroscopic formation, and radioactive ^{40}K contents (Tsoulfanidis et al. 2015). General overview of scintillation detector with a counting system is shown in the Figure 2.9 below.

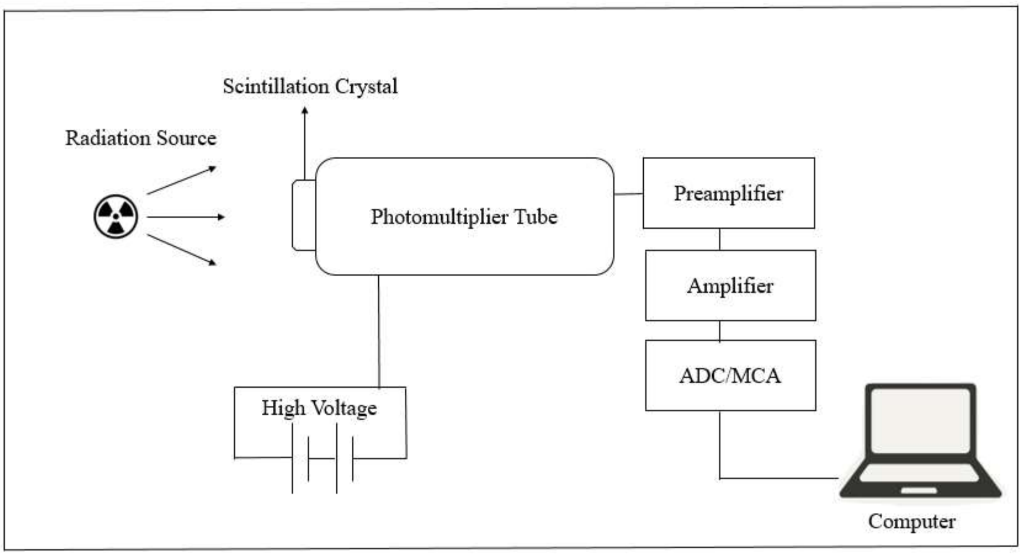

Figure 2.9 Schematic diagrams for scintillation detector with counting system

2.6.3 Digital flat panel radiography detectors

X-ray beam interacted and attenuated by the object is come to flat panel detector. These attenuated photon beam is converted to electrical charges and these electrical charges are converted to signals with help of the electronic circuits in the flat panel detector. The signal pulses are processed then object image is created. There are two types of flat panel detectors which are direct and indirect systems (Bushberg 2012). An indirect system that consists of a scintillation crystal, photodiode and TFT (thin film transistor) is used with in the conventional x-ray unit in order to analyze homogeneity and attenuation properties of prepared samples in the petri dishes. The indirect system that used in this thesis study contains amorphous silicon thin film transistor based CsI scintillation crystal.

2.7 Software Used in the Thesis Study

2.7.1 ImageJ software

ImageJ is open source software which operates based on Java platform. This software is used for scientific image analysis. The program is developed by National Institutes of Health and the Laboratory for Optical and Computational Instrumentation (https://imagej.nih.gov/ij/index.html-2019). Although ImageJ is able to open and read plenty of image formats; it was used for analyzing of DICOM formatted images which were taken by conventional x-ray unit and DRX-1C digital detector. After selection of a circular ROI, for analyzing the prepared samples "analyze and measure" tools were used for determination of pixel values for in each sample. ROI selection and analyze menu is visualized as the following figure;

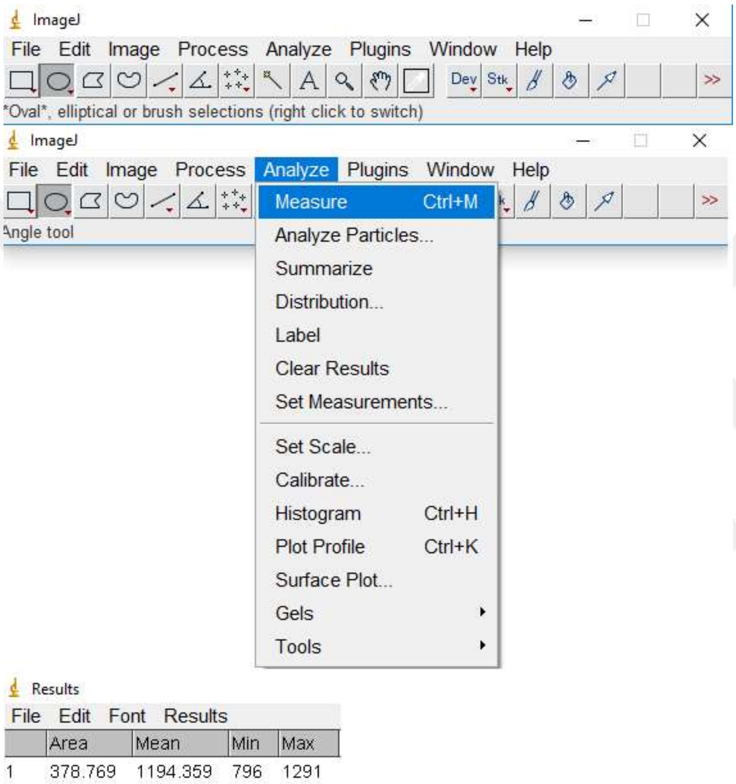

Figure 2.10 Steps of analyzing DICOM image on ImageJ program

3. MATERIALS AND METHODS

3.1 Systems Used in the Thesis Study

3.1.1 Mammographic x-ray system

Device Instillation Date:	2010
Manufacturer:	General Electric Healthcare
Model:	Alpha RT MGF-101
Generator:	Tuned High Frequency(THF)
	Frequency: 40-100 kHz
	kV range: 20-35kV, tolerance ± 1.5 kV
	mA range: Focus 0.3mm 70-100mA
	Focus 0.1mm 20-30mA
	Maximum mA output: Focus 0.3mm 150mA
	Focus 0.1mm 45mA
	mAs range: Focus 0.3mm 2-500 mAs
	Focus 0.1mm 4-300 mAs
Filtration:	0.03mm Molybdenum
	0.025mm Rhodium
X-Ray Tube:	Varian M113SP in B112 Housing
	Anode type: Rotating dual-angle anode,
	Anode material: Molybdenum alloy
	Anode rotation speed: 3000 rpm (50Hz)

Figure 3.1 General Electric Healthcare Alpha RT Mammography Divice (http://www.gme.com.lb/pdf_products/Alpha%20RT.pdf-2019)

3.1.2 Calibration x-ray system

Device Instillation Date:	2010
Generator:	Italray, Pixel HF
	Frequency: 50/60 kHz
	Maximum power: 50 kW
	kV range: 40-150 kV in radiography mode
	40-125 kV in fluoroscopy mode
	mA range: 25-600 mA in radiography mode
	0.5-6 mA in fluoroscopy mode
X-Ray Tube:	Varian, RAD-21
	Focal Spot Size: 0.6 mm- 1.2 mm
	Anode material: Tungsten
	Anode angle: 12
Voltage Divider:	GiCi-PM, Model 2000
	Voltage range: 0-150 kV
	Division ratio: 10000:1 or 1000:1
	Impedance: 1 MΩ or 10 MΩ

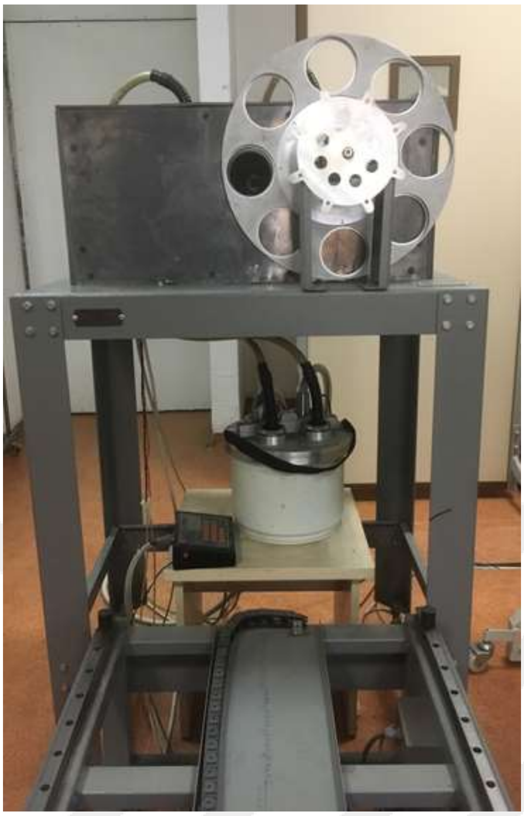

Figure 3.2 Ankara University Calibration Laboratory X-ray System

3.1.3 Conventional x-ray system

Device Instillation Date:	2007
Manufacturer:	General Electric Healthcare
Model:	Silhouette VR
Generator:	GE three phase generator
	Frequency: 50/60 kHz
	Maximum power: 50 kW
	kV range: 40-150 kV
	mA range: 25-600 mA
X-Ray Tube:	Varian, RAD-21
	Focal Spot Size: 0.6 mm- 1.2 mm
	Anode material: Tungsten
	Anode angle: 12

Figure 3.3 General Electric Healthcare Silhouette VR Conventional X-ray System

3.1.4 ^{137}Cs irradiation unit

Device Instillation Date: 2009

Manufacturer: Hopwell Designs, Inc., G10 Gamma Beam Irradiator

1st Radioactive source ^{137}Cs

Activity of 1st source 47 mCi (as of 14.05.2009-reference date)

2nd Radioactive source ^{137}Cs

Activity of 2nd source 8.58 Ci (as of 14.05.2009-reference date)

Attenuator Plates: 3 pieces of 19.4 mm thick lead plate

Collimator: ISO Collimator, 17º

Figure 3.4 Hopewell Designs, Inc., G10 Gamma Beam Irradiator(^{137}Cs) and Radiometric Bench (http://nukbilimler.ankara.edu.tr/kalibrasyon-ve-olcum-laboratuvari/-2019)

3.2 Detectors Used in This Thesis Study

3.2.1 Ion chambers

Table 3.1 Detector specifications used in the thesis

Detector Name	Model	Operating Voltage	Dose Range	Dose Rate Range	Energy Dependence
PTW 28 cm^3 Spherical Ion Chamber	TM 32005	+400 Volts	2.2µGy-23.8mGy	12.9µG/min-65Gy/min	Between 48 keV- ^{60}Co ≤ ± %5

Table 3.1 Detector specifications used in the thesis (Continued)

PTW 30 cm^3 Cylindrical Ion Chamber	TM 23361	+300 Volts	2.0µGy-20 mGy	12µGy/min-56Gy/min	Between 40 keV- ^{60}Co ≤ ± %4
Radcal 6 cm^3 Mammography Probe	10x6–6 S/N 04/0346		100 nGy-700 Gy	600nGy/min-11.4Gy/min	Between 30keV-1.33Mev %5

3.2.2 Ion chamber electrometers and digitizers

In this thesis study; two PTW brand UNIDOS webline model electrometers are used with PTW brand ion chambers in both calibration laboratory and ^{137}Cs Irradiation unit as a monitor. Electrometer is an electronic device which converts electrical signals or voltage to dose or dose rate value. In addition, one Radcal brand Accu-Gold + model digitizer is used with Radcal ion chamber in which used in mammography laboratory.

3.2.3 Digital detector

The digital detector in the conventional x-ray system allows the display of the desired material. Carestream brand and DRX-1C model digital detector was used in this system. This detector contains caesium iodide scintillation crystal. Received images are transferred to computer via wireless connection. Images are used for homogeneity test of the produced material, analyse of the images are made in the computer program ImageJ.

3.2.4 Sodium iodide detector

Gammatech brand sodium iodide (NaI(Tl)) detector is used to collecting spectrum from PbO powder against impurities and ^{210}Pb gamma radiation release. The type of the detector is NDI-65/50, tube diameter is 65 mm, PMT diameter is 53 mm and crystal diameter is 50 mm (http://www.gammatech.hu /downloads/eng/datasheets/nuclear.pdf-2019).

3.2.5 Detectors used in kV measurement

For kV accuracy; Radcal brand, AGMS-D+ model diagnostic energy range kVp meter and AGMS-M+ mammographic energy range kVp meters are used with Radcal brand Accu-Gold + model digitizer.

3.3 Material Properties of Sample Components and Preparation Steps

3.3.1 Technical grade lead oxide powder

Thused lead oxidePbO has 9.53 $g{\cdot}cm^{-3}$ density, 223.2 $g{\cdot}mol^{-1}$ molar mass. Its boiling point is 1470 °C. Its purity is greater than 99%.

3.3.2 Two component epoxy resin and hardener

The elemental composition of epoxy resin and hardener mixture is 68.25% C, 14.67%O, 7.91%H, 4.56%N, 1.11% Ca, 1.48%Cl, 0.456% Zn and 1.56% Si in weight (Yücel et al, 2019). Density of the epoxy mixture is measured as 1.14 $g{\cdot}cm^{-3}$ by using Radwag brand PS X2 model density measurement kit, which based on Archimedes' principle.

3.3.3 Laboratory mixer with speed adjustment

Mtops brand and MS 5010D model laboratory stirrer was used to get homogenous mixture of lead oxide epoxy matrix and hardener. Maximum round per minute of the stirrer is 1000.

Figure 3.5 Mtops Brand Laboratory Stirrer

3.3.4 Sample preparation steps

Preparation of samples was made in the mammography laboratory bench which includes laboratory mixer with speed adjustment, electronic precision balance, beakers and silicone moulds. Each sample contains highly pure lead oxide and epoxy resin with its hardener in different ratios. Two component epoxy resin is nearly transparent to ionizing radiation but it is useful as a matrix of lead oxide which is in the form of the powder. Precision scales are used to measure the weights of epoxy, hardener and lead oxide powder separately. After weight measurement, the sample components are placed in the beaker and mixed with an electrical laboratory mixer at 70 rpm for 20 to 25 minutes. After required mixing time, mixture moulded to 10 cm x 10 cm square moulds and 6 cm radius petri dishes. 24 samples were prepared in petri dishes for homogeneity testing on sample images and their attenuation properties were tested in the mammography energy range. 19 samples were prepared in 10 cm x 10 cm silicone square moulds with different thicknesses for determination of HVL and Lead equivalence of the material in radiological and industrial energy ranges.

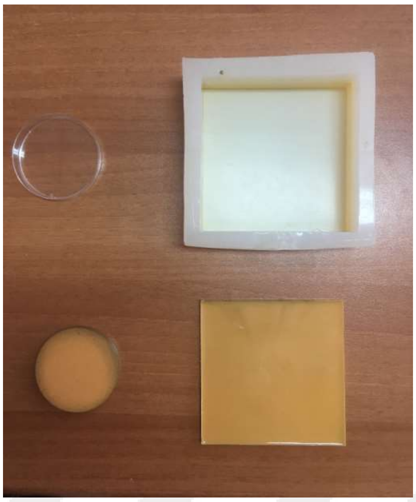

Figure 3.6 6 cm radius (left) and 10cmx10 cm square (right) samples and moulds

3.3.5 Determination of density of samples

It is very significant to determine density of the samples for homogeneity and other material properties such as mechanical and physical, for this purpose Radwag brand electronic precision balance and density measurement kit (PS X2 model) are used. This measurement kit works up on to Archimedes principle.

Figure 3.7 Radwag PS X2 Density Measuring Kit (https://radwag.com/en/kit-128-densitydetermination-kit,w1,B97,103-122#3-2019)

3.3.6 Charpy impact test for samples

By using Instron brand, Dynatup 9250 HV model charpy impact test machine, impact of the samples was determined by Middle East Technical University, Mechanical laboratory. This test is applicable for plastics, ceramics, composites and low energy metal alloys. 10 cm x 10 cm samples are necessary for this test, different sample thicknesses up to 2 cm are applicable.

3.4 Determination of Attenuation Properties of Shielding Samples in Mammography System

This system is specific to breast imaging by using X-ray between 20 to 35 kVp energy ranges. X-ray energy alters with breast thickness, breast composition etc., but it is known that X-rays produced in mammography systems can reach two-meter distance in air. However, it is still significant to determine shielding requirement of the mammography rooms' by using new type materials other than sheet lead or aluminium. In this thesis, the system was used for determination of half value layer (in mm Al) between 20 to 30 kVp energy ranges with rhodium filter and also attenuation tests for samples prepared in petri dishes. X-ray produced with rhodium filter is more penetrable since its mean energy is greater with respect to molybdenum filter. If a sample attenuates X-ray with rhodium filter, it also attenuates with molybdenum filter. For determination of HVL value with different kVp values and irradiation of the samples, the used geometry is installed below (Figure 3.8). In this configuration, Distance A is the distance between anode focus to detector entrance (65.5 cm) and Distance B is the distance between object (sample or aluminium sheet) and detector entrance (7.5 cm)

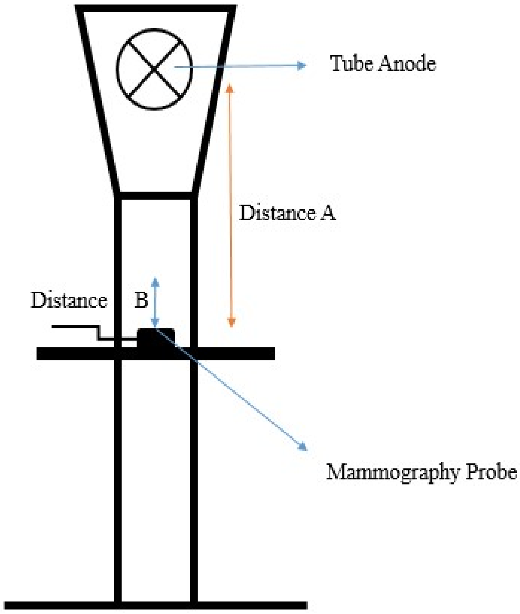

Figure 3.8 Measurement Geometry in Mammographic System

3.5 Determination of RQR Beam Qualities Specified in IEC 61267 Protocols

RQR2, RQR3, RQR4, RQR5, RQR6, RQR7, RQR8 beam qualities were obtained by using following measurement geometry (Figure 3.9). The distance between focal point of the tube to detector was 100 cm. The distance between focal point of the tube to aluminium sheets were 20 cm. Absorber filters are 99.90 % pure aluminium. Measurements were made up with PTW brand TM32005 model 28 cm^3 spherical ion chamber. In calibration X-ray system there is no collimation system operates with visible light so for adjustment of irradiation area; 30 cm x 30 cm a phosphorus layer was used in the dark room. While doing adjustments in manual collimation system and phosphorus layer, 2mm lead shielding and lead glass were used for radiation protection purposes. As a result, irradiation area was fixed to 10 cm x 10 cm.

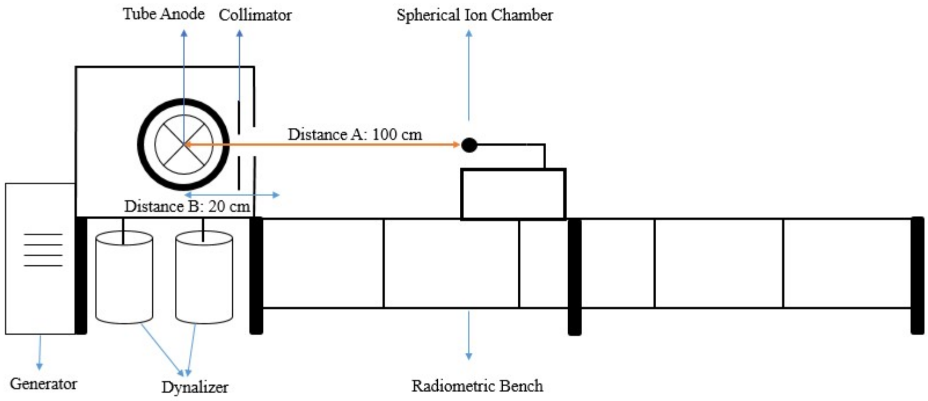

Figure 3.9 Measurement geometry for RQR beam qualities

3.5.1 Determination of 1st HVL, 2nd HVL and homogeneity factor of the system

First HVL or just HVL is the thickness of the material in millimetres which reduces the dose rate (μGy/s) to half of its initial value. In here, absorber material is %99.9 pure aluminium sheets. Second HVL is obtained by subtraction of the first HVL from the thickness of the material that reduces the dose rate (μGy/s) to quarter of its initial value. Homogeneity factor is the algebraic ratio of first HVL to second HVL. During the determination of HVL values, total dose (μGy) values were used instead of dose rate(μGy/s) to avoid being affected by low dose rate values. Between 40 kVp to 100 kVp by steps of 10 kVp's, in each kVp value by adding aluminium sheets dose values were recorded. HVL values are obtained from the graph in which the recorded dose values are plotted with increasing aluminium thicknesses.

3.6 Determination of Attenuation Properties of Samples in Calibration System

10cm x 10cm samples which moulded in different PbO/epoxy matrix ratios were used with different sample thicknesses for HVL measurement of the samples, lead equivalence of the samples and x-ray attenuation ratios of samples. For the measurement, the irradiation geometry is configured as described in the standard (Figure2.5). In four categories of the samples (%60, %50, %40 and %30 with respect to PbO ratios in weight) X-ray attenuation properties are identified individually.

3.6.1 Determination of transmission curves in 80 kVp and 100 kVp standard beam qualities

At 80 kVp and 100 kVp standard beam qualities were determined as explained in IEC 61331-1. In these X-ray beam qualities, for obtaining transmission curves lead sheets (0.5mm, 1mm, 1.5mm) were placed on a distance respectively as shown in Figure 2.5. After that, lead thickness versus dose rate graph is plotted and transmission curve equation is obtained.

3.6.2 Determination of lead equivalence and HVL of samples 80 kVp and 100 kVp standard beam qualities

The samples were placed in a distance as shown in the Figure 2.5, then irradiated with 80 kVp and 100 kVp standard x-ray beams respectively, the recorded dose rate values are used in transmission curve equation. As a result, lead equivalent of the sample was obtained from the fitted curve. For obtaining the HVL of the samples in 80 kVp and 100 kVp X-ray beam qualities, the samples loaded with same lead oxide ratios were irradiated with increasing sample thicknesses, so decreasing dose rates were measured. After that, dose rate vales versus sample thickness were plotted in a graph and HVL value for the corresponding sample is calculated.

3.7 Determination of Attenuation Properties of Samples in ^{137}Cs Irradiation Unit

In this part of the study, it was used ^{137}Cs gamma irradiation source. For measurements the configuration which seen in the Figure3.10 is constructed. Distance A is the distance between ^{137}Cs source to detector. And Distance B is the distance between detector to sample. This unit was used to determine HVL of the samples and lead equivalence of the samples as in the industrial energy range.

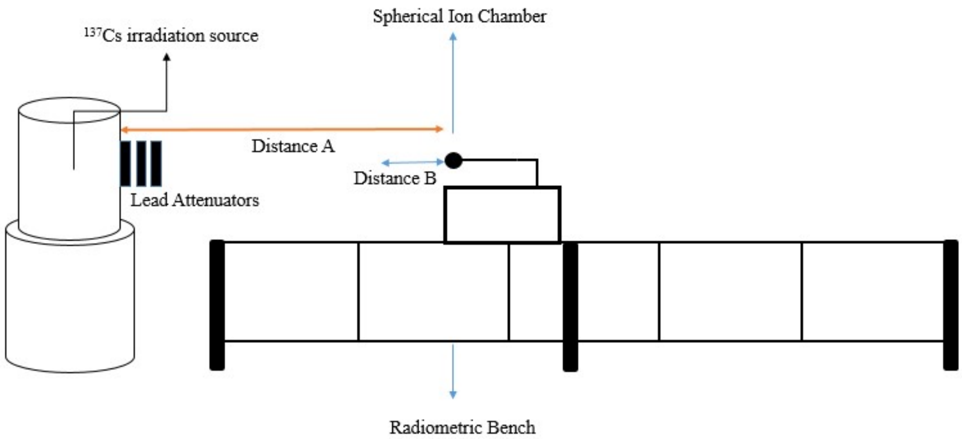

Figure 3.10 Measurement Geometry for 662 keV photon beam HVL and Lead Equivalences in ^{137}Cs irradiation unit

3.7.1 Determination of transmission curves in ^{137}Cs irradiation unit

In the ^{137}Cs source, for obtaining transmission curves lead sheets with different thicknesses were placed on a Distance B respectively as shown in Figure 3.10. After that, lead thickness and dose rate curve graph was plotted and transmission curve equation saved.

3.7.2 Determination of lead equivalence and HVL of samples with ^{137}Cs irradiation unit

The samples were placed in Distance B as shown in the Figure 3.10, and then irradiated with ^{137}Cs source; dose rate values are saved and put into transmission curve equation. As a result, lead equivalent of the sample was obtained from the curve equation. For obtaining the HVL of the samples in ^{137}Cs source, samples with same lead oxide ratios were irradiated with increasing sample thicknesses, so decreasing dose rate is recorded. After that, dose rates versus sample thicknesses were plotted in a graph and HVL value for the corresponding sample is calculated.

4. RESULTS

4.1 Material Analysis

4.1.1 Gamma spectroscopic analysis of PbO with use of NaI(Tl) detector

Lead oxide powder was placed in a cylinder baker and counted on the detector surface to measure possible radioactive impurities in the PbO powder before its use as a substitute. Since there were no observable gamma peaks, PbO powder was used for sample preparation.

4.1.2 Physical properties of prepared samples

Two different molds were used for sample preparation. For industrial and radiological energy ranges 10 cm X 10 cm square shaped molds were used and 19 different samples were prepared the properties of samples are given in Table 4.1 below. For mammographic energy range tests and determination of the sample homogeneity in the conventional x-ray unit 6 cm radius circular shaped petri dishes were used and 24 samples were prepared in 8 groups. The properties of circular shaped samples are given in the Table 4.1

Table 4.1 10 cm X 10 cm square mold samples

Mold Type: 10 cm X 10 cm Square Mold			
Lead oxide percentage	**Number of Sample**	**Net mass(g)**	**Sample Thickness (mm)**
60% PbO	1	71.22	3.17
	2	97.84	3.90
	3	133.58	5.80
	4	243.80	11.01
	5	628.97	28.30
50% PbO	1	61.58	3.19
	2	74.24	4.09
	3	131.78	7.11
	4	202.45	10.75
	5	524.51	28.50
40% PbO	1	57.13	3.46
	2	64.35	4.04
	3	163.92	10.10
	4	241.60	14.76
	5	463.94	28.60

Table 4.1 10 cm X 10 cm square mold samples (Continued)

30% PbO	1	49.78	3.54
	2	82.81	5.45
	3	113.09	7.72
	4	406.70	28.50

Table 4.2 6 cm circular mold samples

Mold Type: 6 cm Radius Petri Dishes			
Lead oxide percentage	**Number of Sample**	**Net mass(g)**	**Sample Thickness (mm)**
50% PbO	1	25.56	5.03
	2	23.98	4.84
	3	24.40	4.94
40% PbO	1	24.09	5.30
	2	23.94	5.23
	3	23.97	5.25
30% PbO	1	24.00	6.00
	2	23.97	6.01
	3	24.00	5.99
20% PbO	1	24.45	6.91
	2	24.20	6.80
	3	24.04	6.85
15% PbO	1	23.89	7.26
	2	23.97	7.20
	3	24.01	7.25
10% PbO	1	23.94	7.38
	2	24.09	7.42
	3	24.03	7.38
5% PbO	1	24.08	7.71
	2	23.99	7.72
	3	24.01	7.68
2% PbO	1	24.13	7.84
	2	23.99	7.82
	3	24.21	7.85

4.1.3 Physical density results

Density of the samples that prepared for industrial and radiological energy ranges were determined with using a density balance kit which works up on to Archimedes principle. The determinations were done in both ethyl alcohol and distilled water. And results are given in the Table 4.3

Table 4.3 Density of samples

Lead oxide percentage, (%)	Density in water-ρ, (g·cm^{-3})	Density in ethyl alcohol –ρ, (g·cm^{-3})	Difference, (%)
60% PbO	2.221	2.196	1.14
50% PbO	1.827	1.855	-1.51
40% PbO	1.693	1.707	-0.82
30% PbO	1.426	1.420	0.42

4.1.4 Impact strength test results

As a result of the test made by METU mechanical laboratory, 0.5mm 60% samples are resistant to 11-12 joule force. Test are done for 50% and 60% samples results are consistent, but not enough to compare between 50% and 60%.

4.1.5 Homogeneity and reproducibility test with conventional x-ray unit

Table 4.4 Homogeneity and reproducibility test with conventional x-ray unit

Lead oxide percentage	Sample number	Maximum pixel values	Average of maximum pixel values	Background pixel values
50% PbO	1	477	507±27	2289
	2	519		
	3	526		
40% PbO	1	671	684±14	2283
	2	698		
	3	682		
30% PbO	1	903	915±12	2290
	2	916		
	3	926		
20% PbO	1	1256	1266±12	2295
	2	1263		
	3	1280		
15% PbO	1	1461	1460±11	2292
	2	1471		
	3	1449		
10% PbO	1	1689	1686±10	2296
	2	1675		
	3	1694		
5% PbO	1	1895	1924±26	2294
	2	1934		
	3	1944		

The homogeneity of the prepared samples can only be determined by photon stopping capabilities. For this purpose, the images were taken in the radiologic energy range (50 kVp) and the pixel values were determined with the help of the ImageJ Program. For this determination circular shaped samples were used and the results are represented in the Table 4.4.

Figure 4.1 shows the trend of average value of maximum pixel counts given in Table 4.4 with increasing PbO amount in the produced samples. As expected, the maximum pixel values are decreasing linearly with increasing the percentage PbO in molded samples. Moreover, identically prepared samples show nearly same photon attenuation behavior.

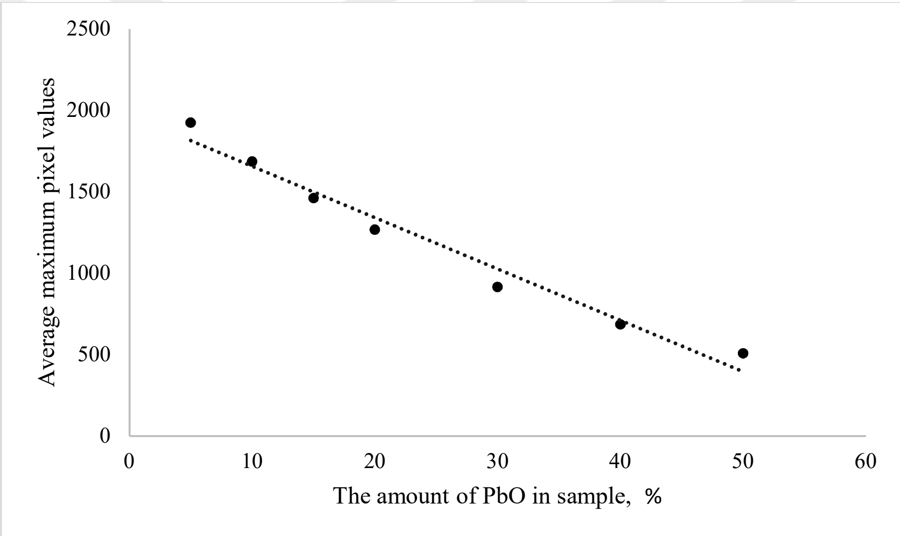

Figure 4.1 The amount of PbO % versus average of maximum pixel values

4.2 Results for Mammographic Energy Range

4.2.1 Half value layers of the mammography system with rhodium filter

The mammography device can be used with molybdenum anode and molybdenum filter (Mo/Mo) or molybdenum anode rhodium filter (Mo/Rh) combinations. However, because the x-ray generated by the rhodium filter is harder, the molybdenum anode rhodium filter (Mo/Rh) was used to test the shielding material in the mammographic energy range and the HVL of the system was measured in different kVp's and 80 mAs.

Table 4.5 Dose (mGy) values measured against increasing aluminum thickness in the range of 23-30 kVp and the HVL values calculated according to them

mm Al	Dose (mGy) at 23 kVp	Dose (mGy) at 25 kVp	Dose (mGy) at 28 kVp	Dose (mGy) at 30 kVp
0	2.84	4.14	6.16	7.90
0.1	2.21	3.38	5.19	6.57
0.2	1.78	2.77	4.26	5.29
0.3	1.45	2.23	3.60	4.69
0.4	1.21	1.87	3.07	3.99
0.5	-	-	2.65	-
0.6	0.84	1.35	2.28	2.95
0.7	-	-	1.99	-
0.8	0.60	0.97	1.72	2.25
1	0.44	0.73	1.35	1.71
1.2	0.32	0.56	1.06	1.32
1.5	0.21	0.38	0.73	0.94
HVL (mm Al)	**0.34 mm**	**0.38 mm**	**0.42 mm**	**0.43 mm**

4.2.2 Transmission curves with respect to aluminum thickness in mammographic energy range

Transmission curves at 23, 25, 28 and 30 kVp (80 mAs) were obtained and their equations were used to calculate the aluminum equivalent of the prepared samples.

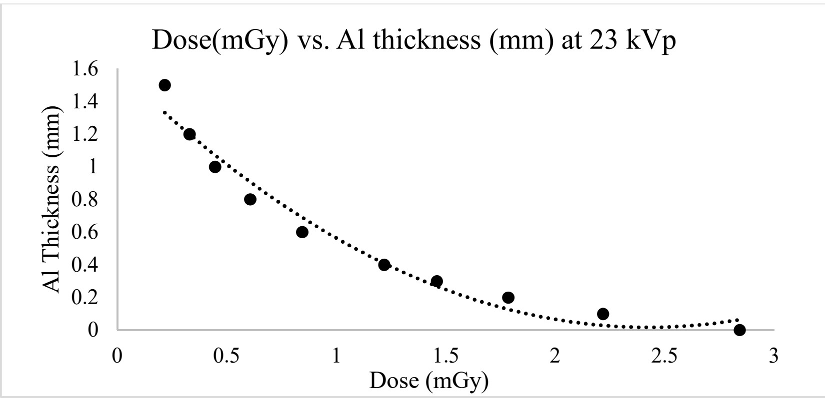

Figure 4.2 Dose vs. aluminum thickness at 23 kVp

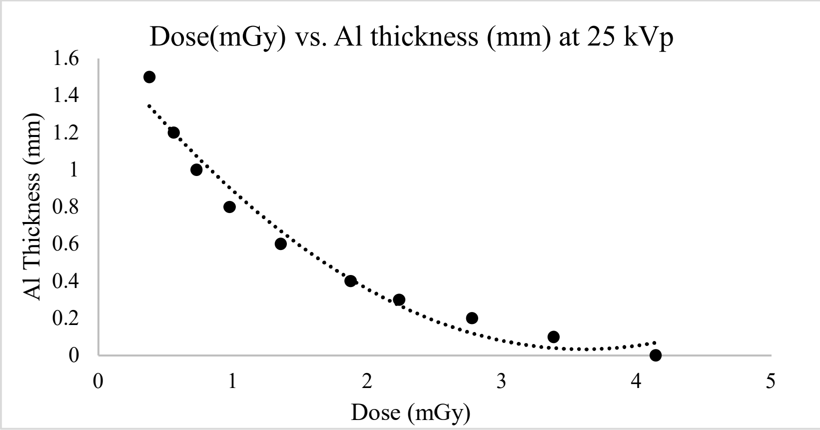

Figure 4.3 Dose vs. aluminum thickness at 25 kVp

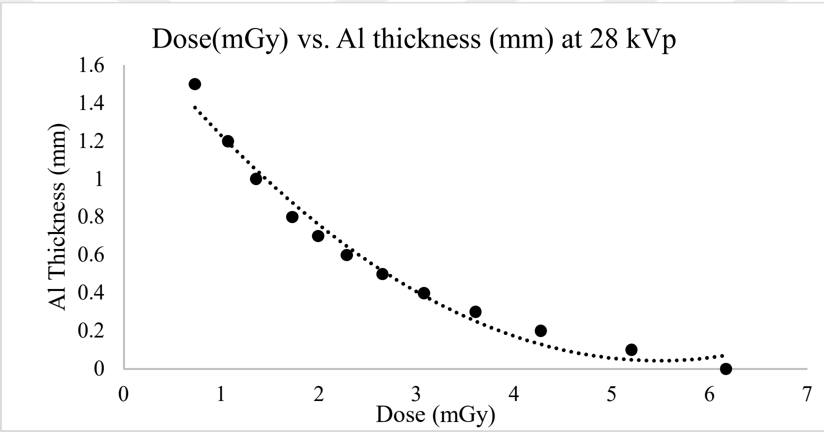

Figure 4.4 Dose vs. aluminum thickness at 28 kVp

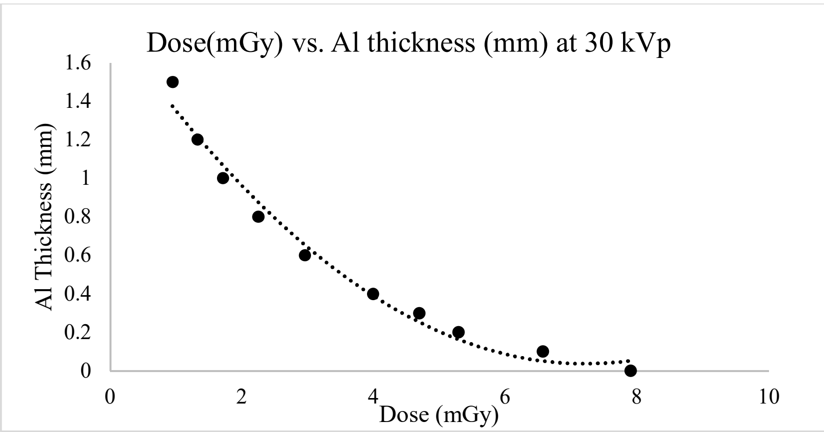

Figure 4.5 Dose vs. aluminum thickness at 30 kVp

4.2.3 Aluminum equivalence of samples in mammographic energy range

Table 4.6 Aluminum equivalence of 2%, 5%, 10% and 15% lead oxide containing samples at 23, 25, 28 and 30 kVp peak voltages and 80 mAs

Lead oxide percentage	Sample number	Filter	kVp/mAs	Detector dose (mGy)	Aluminum equivalent (mmAl)
2%	1	Rh	23/80	0.428	1.04
	2			0.435	1.03
	3			0.435	1.03
2%	1	Rh	25/80	0.675	1.07
	2			0.678	1.07
	3			0.677	1.07
2%	1	Rh	28/80	1.160	1.11
	2			1.125	1.14
	3			1.125	1.14
2%	1	Rh	30/80	1.448	1.14
	2			1.448	1.14
	3			1.446	1.14
5%	1	Rh	23/80	0.087	1.97
	2			0.091	1.94
	3			0.089	1.95
5%	1	Rh	25/80	0.141	2.05
	2			0.149	2.01
	3			0.145	2.03
5%	1	Rh	28/80	0.242	2.22
	2			0.256	2.17
	3			0.249	2.19
5%	1	Rh	30/80	0.319	2.20
	2			0.336	2.17
	3			0.326	2.19
10%	1	Rh	23/80	Not Detectable	-
	2			Not Detectable	-
10%	1	Rh	25/80	0.021	3.18
	2			0.021	3.24
10%	1	Rh	28/80	0.041	3.46
	2			0.038	3.52
10%	1	Rh	30/80	0.056	3.43
	2			0.052	3.48
15%	1	Rh	23/80	Not Detectable	-
	2			Not Detectable	-
15%	1	Rh	25/80	Not Detectable	-
	2			Not Detectable	-

Table 4.6 Aluminum equivalence of 2%, 5%, 10% and 15% lead oxide containing samples at 23, 25, 28 and 30 kVp with 80 mAs (Continued)

15%	1	Rh	28/80	Not Detectable	-
	2			Not Detectable	-
15%	1	Rh	30/80	0.015	4.37
	2			0.015	4.38

4.3 Results for Radiologic Energy Range

4.3.1 kVp accuracy test for calibration x-ray system

Table 4.7 kVp accuracy test with Radcal Accugold+ multisensor detector

Measured kVp	Nominal kVp	Deviation %
40.6	40	-1.50
49.6	50	-0.80
60.1	60	+0.16
69.5	70	-0.71
80.1	80	+0.12
91.2	90	+1.33
101.1	100	+1.10

4.3.2 Determination of total filtration of calibration x-ray system

In order to determine inherent filtration of the x-ray tube, at 60 kVp without additional filtration, HVL value calculated from the curve equation. By using Table 2.1 inherent filtration is interpolated. The HVL at 60 kVp is 1.74 mm Al and corresponding filtration is 1.83 mm Al.

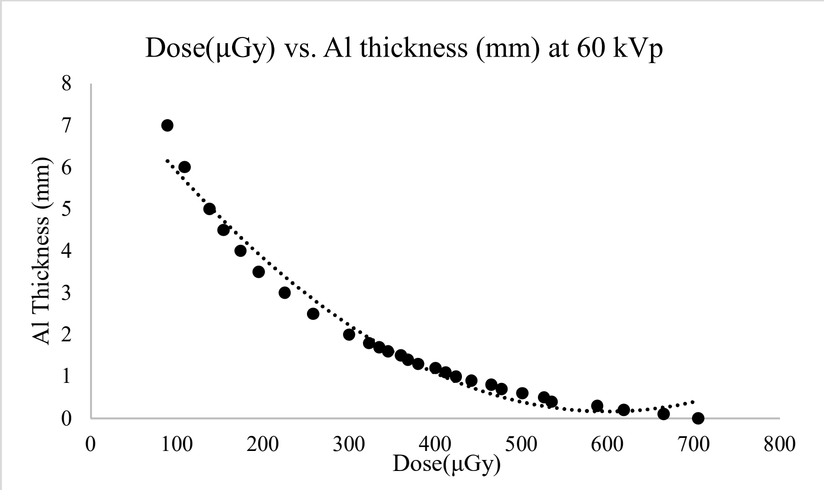

Figure 4.6 Dose vs. aluminum thickness at 60 kVp

4.3.3 RQR x-ray beam qualities

Table 4.8 Obtained RQR beam qualities up to 100 kVp

Radiation Beam Quality	Applied kilo voltage peak (kVp)	1st HVL (mm Al)	Homogeneity factor (h)	Obtained 1st HVL (mm Al)	Obtained homogeneity factor (h)	Added filtration (mm)
RQR2	40	1.42	0.81	1.41	0.81	0.6
RQR3	50	1.78	0.76	1.75	0.77	0.6
RQR4	60	2.19	0.74	2.19	0.89	0.7
RQR5	70	2.58	0.71	2.58	0.81	0.7
RQR6	80	3.01	0.69	3.06	0.77	0.8
RQR7	90	3.48	0.68	3.46	0.73	0.8
RQR8	100	3.97	0.68	3,96	0,92	1

4.3.4 HVL and TVL of the produced samples in radiologic energy ranges

HVL and TVL values of the samples are determined with 80 kVp and 100 kVp beam qualities, for this determination 10cm x10cm square shaped samples which determined in Table 4.1

Table 4.9 HVL and TVL of the produced samples

The percentage Lead oxide loading	HVL (mm sample) at 80 kVp	TVL (mm sample) at 80 kVp	HVL (mm sample) at 100 kVp	TVL (mm sample) at 100 kVp
30%	0.73	3.68	0.93	5.48
40%	0.48	2.34	0.59	3.26
50%	0.27	1.55	0.42	2.53
60%	0.26	1.15	0.36	1.78

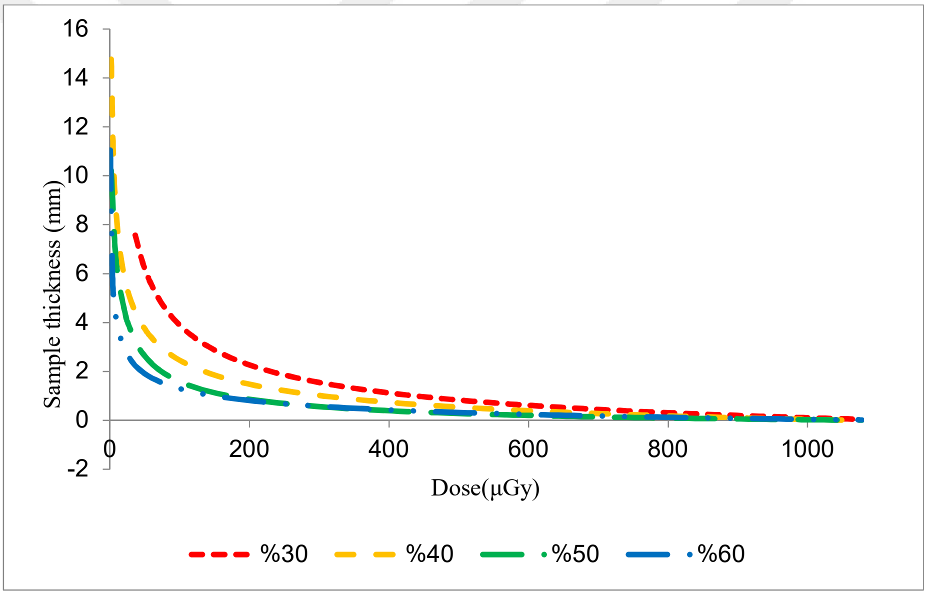

Figure 4.7 Comparison of dose and sample thicknesses with respect to sample ratio (PbO %) at 80 kVp

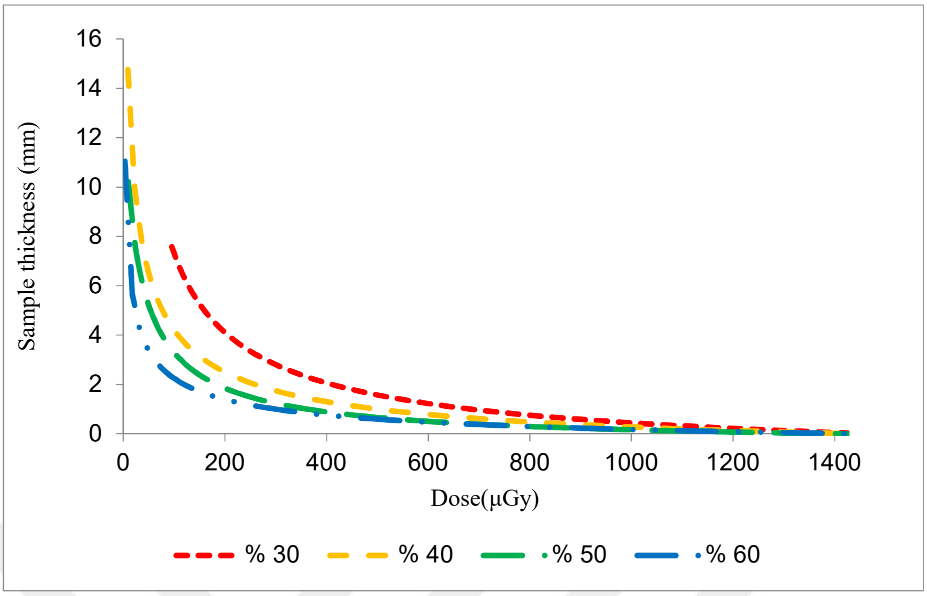

Figure 4.8 Comparison of dose and sample thicknesses with respect to sample ratio (PbO %) at 100 kVp

4.3.5 Transmission curves and lead equivalence in radiologic energy range

Table 4.10 Lead equivalence of the produced samples at 80 and 100 kVp

Percentage Lead oxide loading	**Measured nominal thickness (mm)**	**Lead equivalance (mm) at 80 kVp**	**Lead equivalance (mm) at 100 kVp**
30%	3.54	0.14	0.15
40%	3.46	0.23	0.23
50%	3.19	0.27	0.27
60%	3.17	0.37	0.36
30%	5.45	0.22	0.23
40%	4.04	0.25	0.27
50%	4.10	0.33	0.33
60%	3.90	0.51	0.50

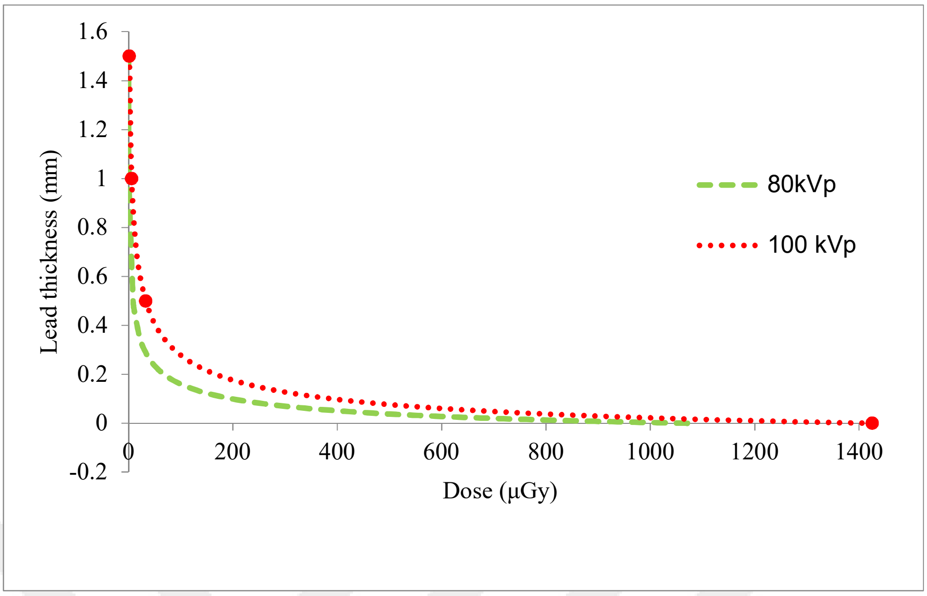

Figure 4.9 Transmission curves up to obtained by using reference lead metal sheet at 80 and 100 kVp

4.4 Results for Industrial (^{137}Cs) Energy Range

4.4.1 Transmission curve with respect to lead thickness in industrial (^{137}Cs) energy range

Transmission curve was obtained from ^{137}Cs (661.66 keV) with respect to 99.9 pure lead sheet thicknesses, and curve was fitted as shown in Figure 4.10.

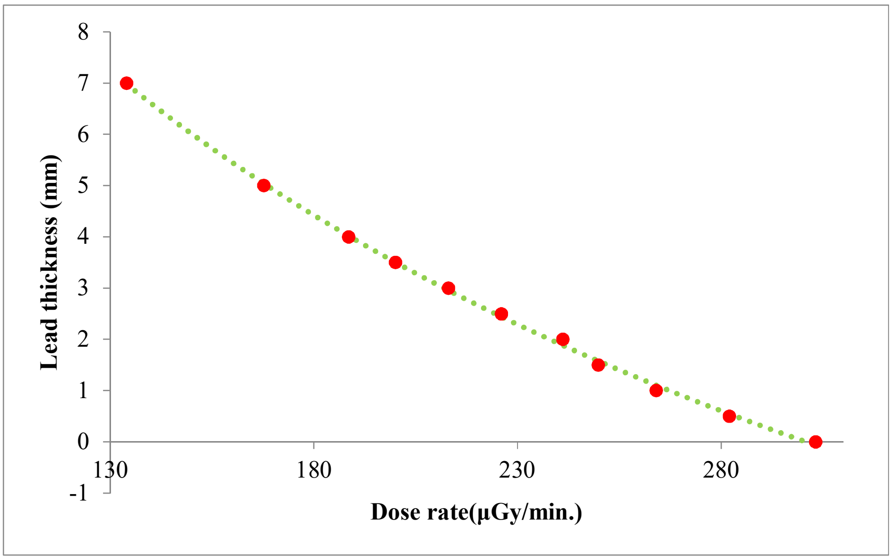

Figure 4.10 Transmission curve up to reference lead metal sheet at 661.6 keV ^{137}Cs

4.4.2 Lead equivalence and HVL of the samples in industrial (^{137}Cs) energy range

Table 4.11 Lead equivalence of samples at 661.6 keV (^{137}Cs)

Percentage Lead oxide loading (%)	Measured Sample Thickness (cm)	Lead Equivalance (mm)	HVL of Samples (cm)	μ calculated (cm^{-1})
60%	1.10	1.51	3.74	0.185
	2.83	4.23		
	3.93	6.31		
50 %	2.85	3.18	4.38	0.158
	3.93	5.17		
	4.64	5.98		
40%	1.01	1.46	5.27	0.131
	2.86	2.54		
	3.87	4.22		
	5.35	5.98		
30%	1.33	1.59	Not measured	Not calculated
	2.85	2.08		
	3.62	3.30		
	4.69	4.16		

5. DISCUSSION AND CONCLUSION

In this thesis, a composite shielding material based on epoxy matrix mixed with powdered lead oxide compound was produced and tested at three different energy ranges. In order to demonstrate the homogeneity of the prepared samples, the x-ray images were taken with the digital imaging detector in the radiological energy range. These images were evaluated in the “ImageJ” image processing program. It was seen that the pixel values decreased in the images in response to increasing lead oxide ratios in the samples. This test was repeated with identical samples and the results were consistent. The physical density of the produced material was measured by using special balance kit working with water and alcohol according to Archimedes principle. As a result, the density of the shielding material ranges from 1.42 $g.cm^{-3}$ (for 30%PbO) to 2.20 $g.cm^{-3}$ (for 60%PbO) based on the ratio of lead oxide/epoxy. The density of the sample containing 60 % lead oxide is slightly lower than ordinary concrete's density.

In the mammography energy range between 20-30 kVp; no dose value measured in the detector for the samples with a thickness of approximately 7 mm containing 20 % lead oxide. 20% lead oxide level and 7 mm thickness are the lower limit for this material in this energy level. Samples produced for values greater than 7mm thickness or lead oxide ratio can be used in the mammographic energy range as a shielding material. This is an expected result because the HVL values in Mo/Mo and Mo/Rh anode filter device, were measured as 0.34 mm to 0.43 mm aluminium. The tests carried out in this energy were repeated with identical samples and the consistency in the results was examined against possible impurities in the material, and the results were observed to be consistent.

In the radiology energy range between 40-100 kVp; in order to obtain the desired x ray beam qualities, HVL values and filtration values were measured at the energies to be studied first. As a result of these measurements, the beam qualities defined in the standards were provided by adding aluminium filters of different thicknesses. After necessary system adjustments, HVL and TVL values of the samples with different lead oxide ratio were measured at 80 and 100 kVp. HVL values for all lead oxide ratios (30% to 60%)are equal or less than 1 mm sample at both 80 and 100 kVp values. TVL values at 80 kVp according to the ratio of lead oxide; it ranges between 1.15 mm sample (for 60%) and 3.68 mm sample (for 30%). At 100 kVp, TVL values varies between 1.78 mm sample (for 60%) and 5.48 mm sample (for 30%). The HVL and TVL values as well as the lead equivalent of the samples were calculated in the radiological energy range at 80 and 100 kVp. For this calculation, the transmission curve generated by pure lead plates of different thicknesses was used. Samples of similar thickness were used to compare the lead equivalent of the samples containing different lead oxide ratios. The lead equivalent results are given in Table 4.10, where the results in 80 and 100 kVp are very similar. This is because the average energy in these two energies is very close to each other. Furthermore, the fact that the samples cannot be mold less than 3 mm is an also reason. Because the reduction curves at 80 and 100 kVp are very close to each other at some points. As a result, conventional x-ray devices are generally shielded with 2- 3 mm lead plates, 4 millimetres of the 60% lead oxide-containing form of this material equals 0.5 mm lead plate. i.e. a 2 cm wall or floor covering made using this material is equivalent to 2.5 mm lead in this energy range.

As an industrial energy range ^{137}Cs was used which has 661.6 keV gamma ray energy. Thicker forms of the samples containing 30% to 60% lead oxide were used in this energy. 99.9% pure lead plates were used to obtain the lead equivalent of the materials in this energy, and the transmission curve was obtained. The lead equivalent for samples containing different percentages of lead oxide in identical thickness (≅2.85 cm) ranges from 2.08 mm (for 30%PbO) to 4.23 mm (for 60% PbO). This result shows that; this novel composite material must be produced in the form of bricks of high thickness in order to attenuate a very high activity gamma source used in the industrial field. In addition to the lead equivalent, the HVLs of the samples were also calculated for this energy, but could not reach the half value for the sample containing 30% lead oxide. The HVL values for other samples were 3.74cm (%60 PbO), 4.38 cm (%50 PbO), 5.27cm (%40 PbO).

In conclusion, in this thesis the suitability of this new composite material as a shielding, based on epoxy, has been tested. After that, to increase the shielding effectiveness of the novel material; it is advisable to try to increase the ratio of lead oxide added into epoxy resin. 60% lead oxide is upper limit for production after this ratio mixture become inhomogeneous. In addition, the resistance of the material to heat exchange, strength and toxicology tests are important for the use. In addition, the use of bismuth oxide, which is much more expensive than lead oxide, is also advisable because it is not toxic.

In future works, it is suggested that epoxy or other similar bonding organics can be mixed form of double element mixtures, rather than single compound (PbO) to produce new type of shielding materials.

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