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GROWTH AND OPTICS CARACTERISTICS OF THE CsI:Li SCINTILLATOR CRYSTAL FOR USE AS RADIATION DETECTOR

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ABSTRACT

Materials capable of converting ionizing radiation into light photons are called scintillators, some have specific efficiencies for certain applications and types of radiation, e.g. gamma, X-ray, alpha, beta and neutrons. CsI:Tl and NaI:Tl crystals are commonly found in the market because they have several applications, but few studies have been done on lithium doped cesium iodide crystal (CsI:Li). The lithium element, in this crystal used as a dopant, is also exploited as a converter for neutron detection, as it has a shock section of 940 barns for thermal neutrons. The study of the CsI:Li crystal is convenient considering the natural abundance of the lithium element with 7.5%, besides the interest in having a low cost national scintillator material with an opportunity to search the response of a detector for different types of radiation. The CsI:Li crystal was grown with molar concentration 10^{-4} to 10^{-1}, using the vertical Bridgman technique. The parameters involved in the growth process were investigated. The transmittance was evaluated in the spectral region from 190 nm to 1100 nm. Luminescence emission spectra for the CsI:Li crystal were evaluated by photometric analysis of the crystal stimulated with a ^{137}Cs (662 keV) source in front of the coupled sample at the monochromator input. The crystals showed of maximum luminescence intensity at the wavelength of 420 nm. The response of the scintillators when excited with gamma radiation of ^{241}Am, ^{133}Ba, ^{22}Na,^{137}Cs, ^{60}Co and neutron radiation from the AmBe source, with energy range of 1 MeV to 12 Mev was evaluated.

1. INTRODUCTION

Ionizing radiation encompasses a broad spectrum of energy and various types of interactions with matter. As a consequence, each detector has its field of use delimited by the type of radiation, energy interval and characteristics of its physical response. Ionizing radiation has been used for many scientific and industrial purposes, which were discovered more than one hundred years ago. Ionizing radiations are invisible to the naked eye, and some of them have a high penetration power against dense matters. In order to use such ionizing radiations, special tools are necessary to detect and visualize. Such tools are often called radiation detectors; there are two types of solid materials that are most commonly used for radiation detectors. One is semiconductors, and the other is luminescent materials know as scintillators. [1,2]

Scintillators are composed by certain materials, which have the properties to emit light when ionizing radiation interaction. The modern physics of scintillators is a broad field of fundamental and applied research dealing with a large variety of materials and applications. Every year the scintillation method finds new applications in high energy physics, nuclear medicine, geophysics, the monitoring of environmental, in devices and systems for security inspection, non-destructive testing, space physics, astronomy, etc. The destination and operation conditions of scintillation equipment determine a broad set of requirements to scintillation elements and materials. [3]

A special place among scintillation materials belongs to inorganic crystals, with the most used as scintillators constituted of alkali metals, in particular alkaline iodides. Inorganic crystals have been the subject of study to be used as radiation detectors since the 1950s. [4,5] Since then, various inorganic crystals have been studied in various fields of scientific research. With the advancement in the development of different energy particle accelerators, electromagnetic calorimeters, medical diagnostic equipment, research in the field of reactors, the need for development and design of detectors suitable for measurement of particles of different LTEs (Linear Transfer Energy) was required. For high-energy particle accelerators it is common to use multiple detectors capable of discriminating the energy of emitted light, making the cost of the sensor one of the most important items to be chosen. These detectors are usually constructed with thousands of scintillating crystals and in some experiments the total volume of the detector may reach more than $1m^3$ [6]. Therefore, due to the need of large scale crystals for detector design, the choice of these crystals should meet the criteria of the simplicity to obtain and assemble, plus feasible costs. These requirements make the CsI based crystals promising for these applications because they meet these requirements and have the advantage of being used as ionizing radiation detectors under adverse operating conditions, which require high mechanical and thermal resistance [7]. The CsI matrix is poorly hygroscopic, its constituents have a high atomic number, are easy to handle, and have a relatively low cost.

Some scintillators are manufactured with neutron active material added to achieve enhanced neutron detection capability. The purpose is to achieve more localized and more rapid detection of neutrons than is possible with gas counters. Gadolinium, boron and lithium are typical materials loaded into the scintillator. Lithium is a very attractive element because it presents natural abundance of 7.5% in ^{6}Li. [8,9] The ^{6}Li is used in this study as a converter for detecting thermal neutrons. The converter is a material with a high probability of interaction with the neutron and high capture cross section. It features cross section of 940 barns, for thermal neutrons. The reaction of thermal neutrons with ^{6}Li used in detectors may be written as:

$$ {}^{6}_{3}Li + {}^{1}_{0}n \rightarrow {}^{3}_{1}H + {}^{4}_{2}\propto \ + 4.79\ MeV \qquad (1) $$

In this reaction, the emitted tritium has kinetic energy of E+ 2.73 MeV 3H, while α particle is emitted with E_α+ 2.05 MeV [2]

In this context, growth and optics characteristics of the CsI crystals doped with Li ions for use as radiation detectors, were investigated. The pure CsI was used for comparison.

2. MATERIALS AND METHODS

Lithium doped CsI and pure CsI crystals were grown using the vertical Bridgman technique. [10, 11] In the process a number of parameters are involved, including the growth velocity, the temperature profile of the furnace, cleaning materials, and the geometric configuration of the material from which the crucible is made are the major. Was used a furnace for crystal growth for starting materials which have a melting point of below 1000 °C. The system is open and was designed to operate with sealed ampoules inside. The starting material used has purity of 99.99%. The molar concentration of the lithium dopant element was 10^{-4} to 10^{-1}.

In order to obtain crystals of good quality, thermal and mechanical stability are need. Mechanical or thermal fluctuations may produce localized over-colds, leading to irregular growth of the crystal. Mechanical stress, due to thermal expansion and contraction, may be reduced by selecting a suitable crucible. Quartz was chosen as the material to be used in the manufacture of crucibles in the process of crystal growth, with the following aspects: (a) thermal and mechanically stable up to, at least, 100 °C above the melting point of the material to be crystallized, (b) chemically inert to the molten material and not influencing the properties of the crystal, (c) resistant to the atmosphere in which is held the crystal growth, (d) resistant to temperature changes while having low thermal conductivity in high temperature gradients.

The crucibles were subjected to a constriction at its top center, where smaller quartz cylinders were placed in order to facilitate the sealing of the same by means of oxy acetylene solder. The other end of the crucibles was molded into a conical shape, so that the initially formed core in this region can serve as a guide for the crystal growth.

The salt of cesium iodide (CsI) and lithium iodide (LiI) were subjected to a dehumidification process under continuous vacuum of the 10^3 Pa and a temperature of 200 °C for 3 h in order to remove residual water, gas, atmospheric and volatile impurities. The dehumidification process involved the following steps: slow heating of the system under continuous vacuum to the temperature of 100 °C (temperature range in which no hydrolysis occurs), kept at this temperature for 1 h. This temperature, still under vacuum, was maintained by heating the system with a heating rate of 25 °C to 200 °C, keeping this temperature for 3 h, to remove the water absorbed chemically.

For the growth of crystals by the Bridgman technique, was used the two zone oven (hot and cold). The crucible containing the CsI and LiI salts was placed in the hot zone of the furnace, with the melt temperature of 650 °C. After completing the salt melting, it was necessary to stabilize the temperature, and the melting stayed under this condition for 10 hours, to ensure the homogenization of the load. Only then, the displacement of the crucible, started towards the cold zone of the furnace at a speed of 1 mm h^{-1}, by means of a displacement system with programmable logic control. During this procedure, it was avoided any vibration or motion of the solid-liquid interface, as this could cause changes in the orientation of the crystal. The temperature was strictly controlled using a microprocessor-based controller. Finally, after 120 h, the crucible cold be busy with the single crystal.

The grown crystals were subjected to heat treatment. In this procedure, vacuum of 10^{-4} Pa and continuous temperature of 350 °C, for 24 h, were employed. The grown crystals were polished with ethylene glycol pA ($C_2H_6O_2$). The polishing of slightly hygroscopic crystals differs from metal polishing or glazing on two aspects: the material has low hardness and

may deteriorate in the presence of moisture. The softness of the crystal means that with polishing in a fast and in short period of time, a considerable layer of material could be removed. The side surfaces were left not polished to enhance internal reflection.

Transparency is a critical factor for scintillators, since the emitted photon with energy in the visible region, needs to be transported efficiently to the photosensor. The transparency may be evaluated by a direct measurement of light transmittance at a wavelength of flicker. Accordingly transmittance tests were performed on samples of CsI:Li crystals with concentrations 10^{-4} M, 10^{-3} M, 10^{-2} M, 10^{-1} M and pure CsI, using a UV-visible spectrophotometer (Shimadzu UV-1601 PC). The measurement system consists of, basically, the light source, a monochromator and a detector. Between these two elements, the sample was positioned. The magnitude of this study was to measure absorbance (A) which is related to the transmittance (T) through the expression:

$$A = \log(1/T) \Longrightarrow T = 10^{(-A)} \quad (2)$$

The spectral coverage was 190 nm to 1100 nm, and the optical path length was 3 mm. The same measurements were performed for the pure CsI crystal for comparasion purposes. A Thermo Spectronic Standars model 333150, identified as 50% with reference 325D296009-1 with defined transmittance and absorbance values, was used before and after reading the samples, in order to confirm the calibration of the equipment.

Luminescence emission spectra for the CsI:Li and pure CsI crystals were evaluated by photometric analysis of the stimulated crystals, with a radioactive source of ^{137}Cs (662 keV) in front of each sample coupled to the monochromator input. The pulses of light from the scintillators were converted into electrical impulses by means of a photomultiplier tube, optically coupled to the output of the monochromator. The associated electronic for analysis consisted primarily of conventional ORTEC nuclear instrumentation electronics. Fig. 1 shows the schematic diagram of the luminescent emission measurement system of the CsI:Li and pure CsI scintillator crystals.

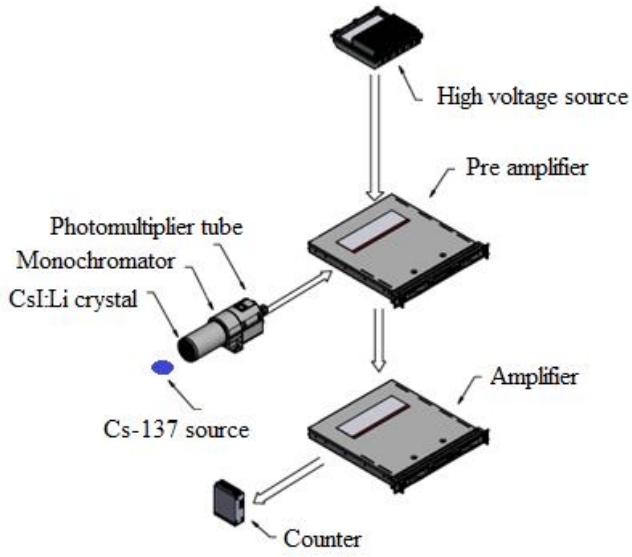


Figure 1: Schematic diagram of the luminescent emission measurement system of the CsI;Li and pure CsI crystals.

In the study of gamma radiation and neutron radiation response, the crystals were machined, polished and coupled directly to the bi-alkaline photomultiplier (model RCA 8575, 21 pins) using a viscosity of 0.5 McStokes silicone grease (Dow Corning) as an optical interface. The pre-amplifier (Ortec model 276), amplifier (Ortec model 450), high voltage source (Ortec 556), multichannel analyzer (ADCAM Ortec model 918A), Phillips oscilloscope (PM3295A 400MHz) and microcomputer. The crystals were prepared 20 mm in diameter and 14 mm thick for both experiments.

Gamma radiation sources were employed, with energies ranging from 59 keV to 1333 keV. The radioactive sources were positioned in the center of the upper face of the crystal. The photomultiplier operating voltage was 2400 V for the detection of gamma rays. The accumulation time in the counting process was 600 s. Fig. 2 shows the diagram of the detection system.

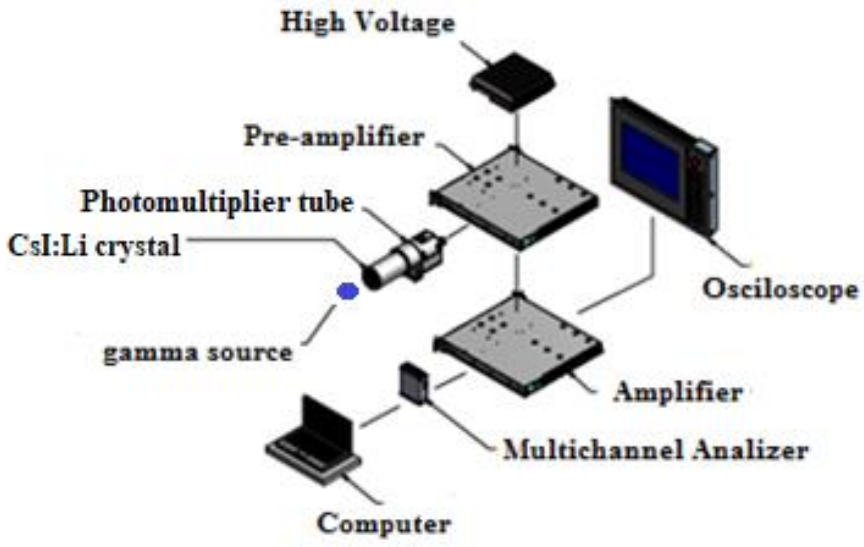
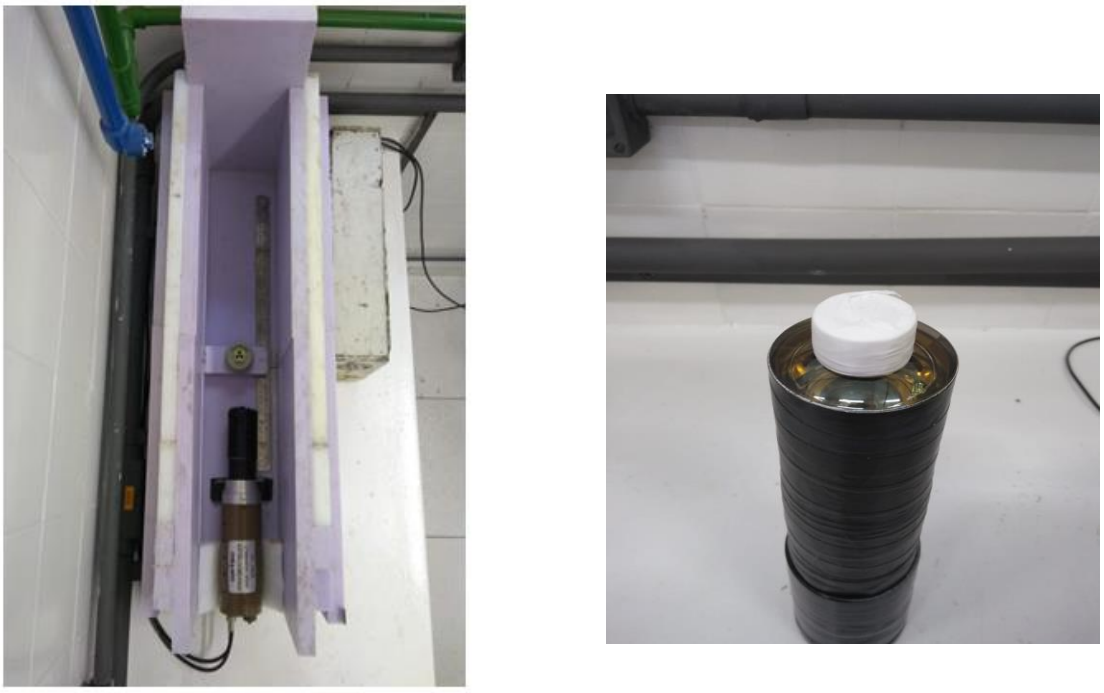


Figure 2 : Schematic diagram of the electronics associated with the CsI:Li crystal coupled to the photomultiplier tube.

In response to neutron radiation, an AmBe source with energy of 1 MeV at 12 MeV, positioned at 70 mm from the crystal was used. The photomultiplier operating voltage was 2400 V. The accumulation time in the counting process was 600 s. The creep was 2.6 x10^6 neutrons / second. Fig. 3 shows: (a) assembled arrangement for the measurement of neutrons, which is formed by a borated polystyrene barrier and paraffin plates for the purpose of protecting the operator and (b) the scintillator crystal optically coupled to the photomultiplier tube .



(a) (b)

Figure 3: Assembled arrangement for neutron measurement, which is formed by a borated polystyrene barrier and paraffin plates (a) and the scintillator crystal optically coupled to the photomultiplier tube (b).

3. RESULTS AND DISCUSSION

In this work, the crystalline crystals were obtained in lithium doped CsI matrix, with a molar concentration of 10^{-4}, 10^{-3}, 10^{-2}, 10^{-1} and pure CsI. Fig. 4 shows a CsI:Li 10^{-3} M crystal: (a) cut crystal in the geometry adopted for this work (b) polished crystal and crystal in the geometric shape of the crucible.

(a) (b)

Figure 4: Scintillator crystal CsI:Li 10^{-3} M. (a) cut crystal in the geometry adopted for this work (b) polished crystal and crystal in the geometric shape of the crucible.

In order to determine the degree of transparency of the scintillator crystals produced, optical transmittance assays were performed in the spectral ranging from 190 nm to 1100 nm.

In all samples of CsI: Li and pure CsI crystals, the transmittance decreased as a function of the wavelength decrease. Fig. 5 shows the transmittance curves of crystals of pure CsI and CsI: Li at various concentrations of the lithium dopant element.

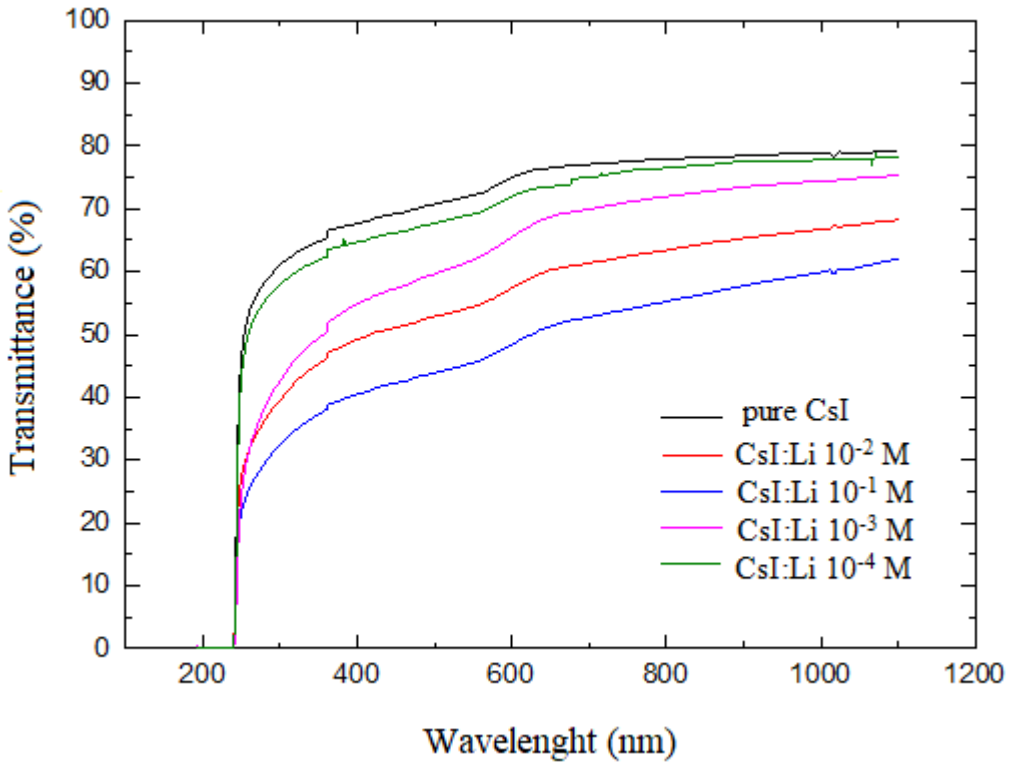


Figure 5: Transmittance curves as a function of wavelength for pure CsI and CsI:Li crystals with thicknesses of 3 mm.

The optical transmittance of pure CsI crystal was 80% at 1100 nm wavelength dropping practically to zero at wavelengths below 240 nm, with values are in agreement with the literature [12]. The CsI:Li 10^{-4} M crystal exhibited behavior similar to pure CsI, with approximately 80% at 1100 nm wavelength and practically zero at wavelengths below 240 nm. The CsI:Li 10^{-1} M crystal showed the lowest transmittance in the lithium concentration range studied, with 60% for 1100 nm wavelength and 40% for 400 nm wavelength.

The CsI:Li 10^{-2} M crystal had an optical transmittance of approximately 65% at 1100 nm wavelength dropping to zero at wavelengths below 240 nm. At wavelengths between 400 nm and 600 nm, the optical transmittance ranged from 50% to 55%. The wavelength range from 400 nm to 600 nm is the area of sensitivity of the quantum efficiency of the photomultiplier tubes.

The optical transmittance of the CsI:Li 10^{-3} M crystal was approximately 73% at 1100 nm wavelength and practically zero at wavelengths below 240 nm. In the wavelength range of 400 nm to 600 nm, region of interest of study of luminescence intensity, the optical transmittance ranged from 55% to 65%.

Significant decrease in transmittance relative to pure CsI crystal was observed as the concentration of lithium in the grown crystals increased. The decrease in transmittance values in the CsI: Li curves can be attributed to the Li absorbance introduced into the crystal lattice.

The luminescence spectra in function of the wavelenght for the pure CsI and CsI:Li 10^{-4} M, 10^{-3} M, 10^{-2} M, 10^{-1} M crystals excited with gamma radiation from a source of ^{137}Cs, are shown in Fig. 6.

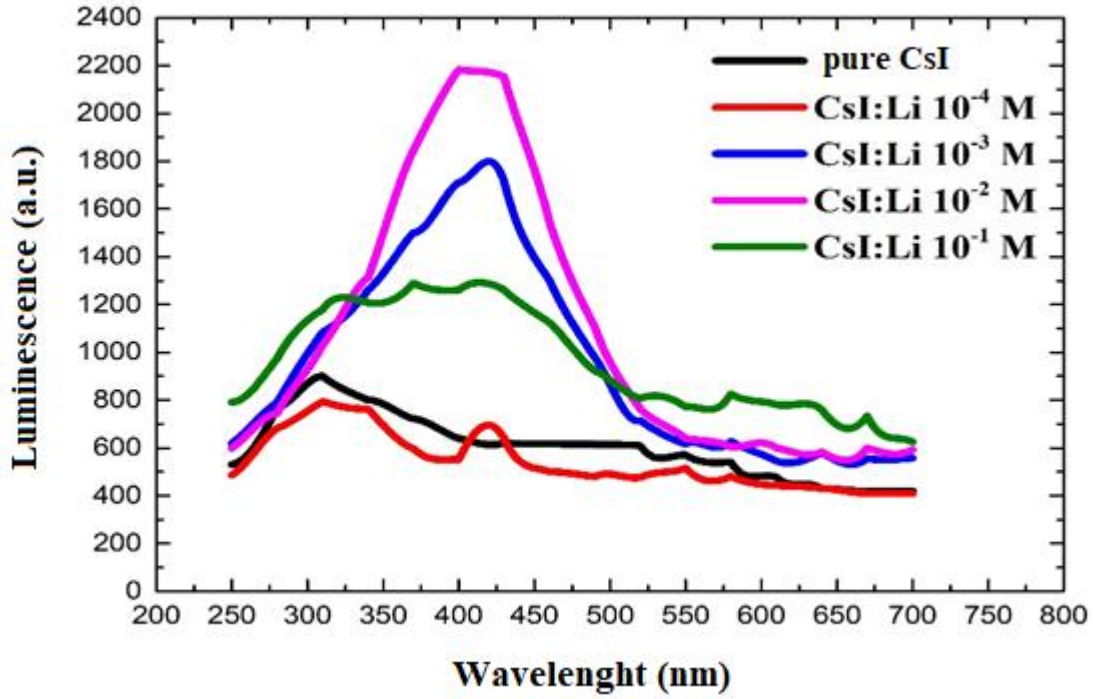


Figure 6: Luminescence spectra for pure CsI and CsI: Li 10^{-4} M, 10^{-3} M, 10^{-2} M, 10^{-1} M crystals using gamma radiation of the ^{137}Cs source.

As can be seen in Fig. 6, the maximum luminescence emission wavelength is characteristic for each crystal. The maximum intensity of luminescence at the wavelength of 320 nm, found for pure CsI crystal, is in agreement with the literature [13]. The origin of this luminescence in the crystal is attributed to the characteristic of the material in its pure form. As shown in Fig. 6, luminescence spectra for the CsI:Li crystals showed light emission peaks at wavelength close to 420 nm for all concentrations of the dopant element. In addition to an intense peak at 420 nm, emission peak with lower intensity near 320 nm was, also, observed in the luminescence spectrum of CsI:Li crystals.

The emission at 420 nm can be attributed to the presence of lithium ions, while the origin of the peak of less intensity at the wavelength around 320 nm could be attributed to the intrinsic luminescence from the pure CsI crystal. As it may be observed in Fig. 6, the CsI:Li 10^{-2} M crystal showed the highest luminescence intensity in the range studied. The CsI:Li 10^{-1} M

crystal showed a significant decrease in luminous intensity when compared to the other concentrations. This decrease in luminescence can be attributed to saturation of the crystal lattice.

Comparing doped crystals and pure crystal, the most significant changes were the significant increase of the luminescence intensity and the peak displacement of the doped crystals to wavelength around 420 nm, in which is the region of greater sensitivity of the tubes photomultipliers (photosensor used in this work), thus presenting an excellent overlap between the luminescence spectrum of the crystals and the quantum efficiency of the photosensor, making these crystals suitable for use as radiation detectors.

The investigation of the crystals as to their performance as gamma radiation detector was carried out using emitting sources with energies in the range of 59 keV to 1333 keV. The radiation sources were placed in contact with the crystals. Individually obtained spectra in response to the gamma excitation undergone by crystals, at the four concentrations produced and by the pure CsI crystal for comparison purposes. Fig. 7 to 11 show the spectra obtained in the detection of gamma radiation.

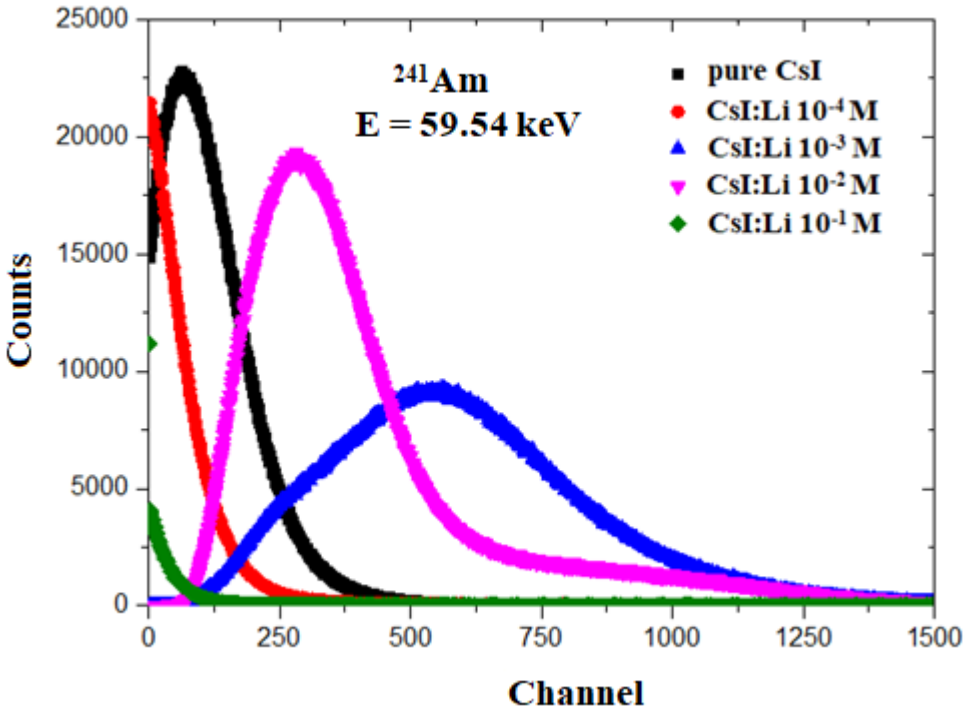


Figure 7: Detection spectra obtained with CsI:Li and pure CsI crystals excited with gamma radiation from a ^{241} Am source.

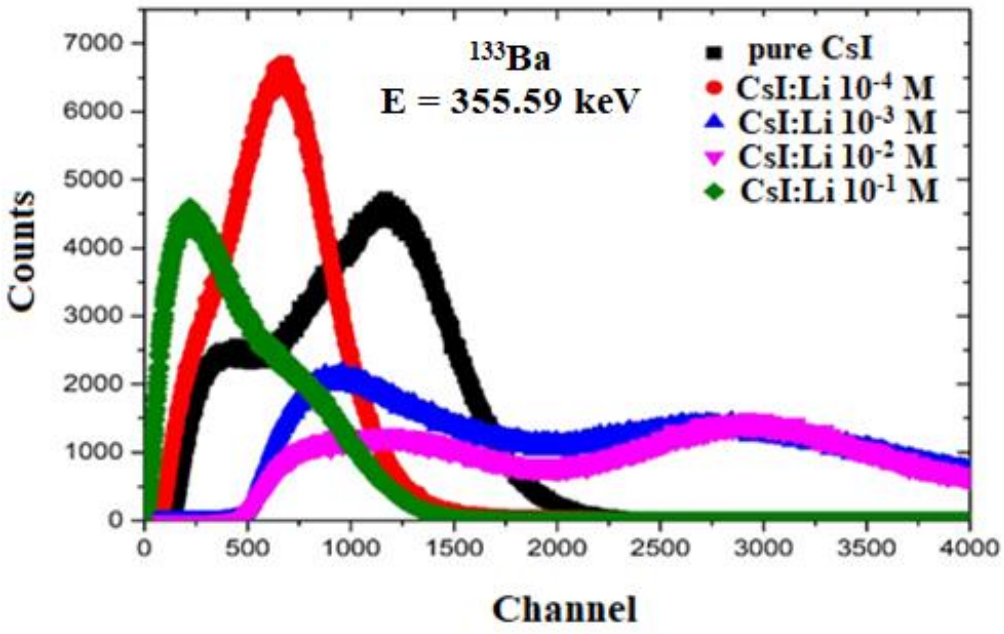


Figure 8 : Detection spectra obtained with CsI:Li and pure CsI crystals excited with gamma radiation from a ^{133}Ba source.

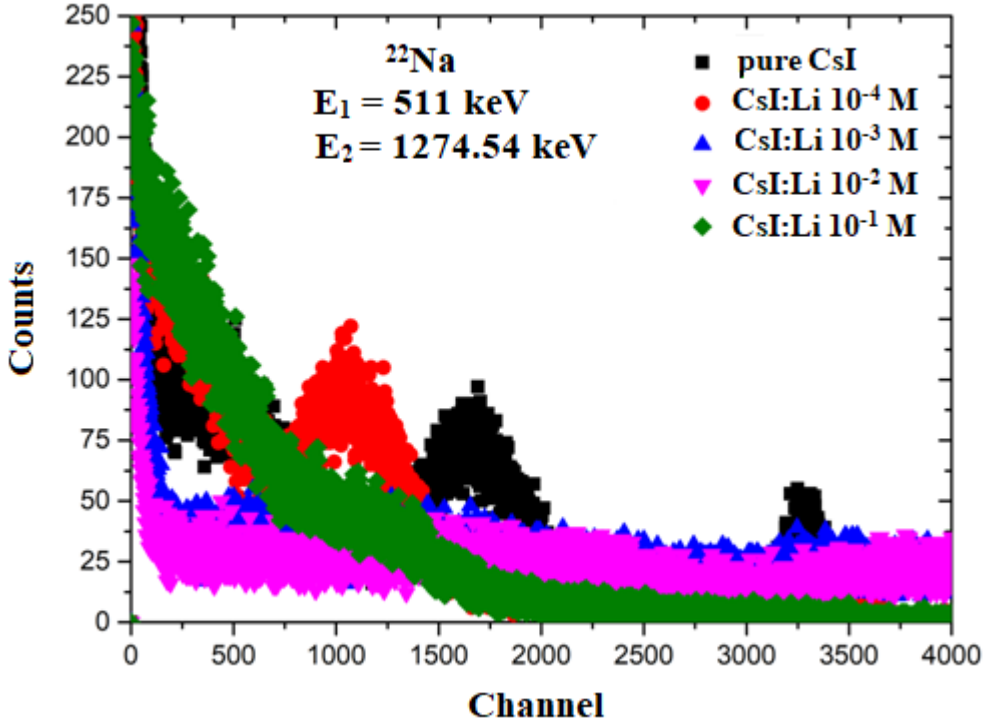


Figure 9 : Detection spectra obtained with CsI:Li and pure CsI crystals excited with gamma radiation from a ^{22}Na source.

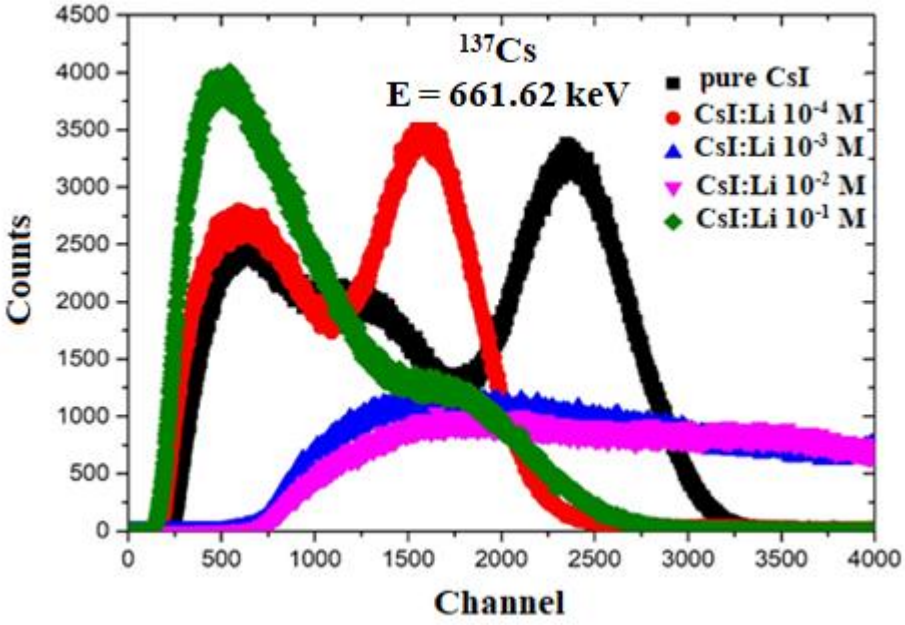


Figure 10 : Detection spectra obtained with CsI:Li and pure CsI crystals excited with gamma radiation from a ^{137}Cs.

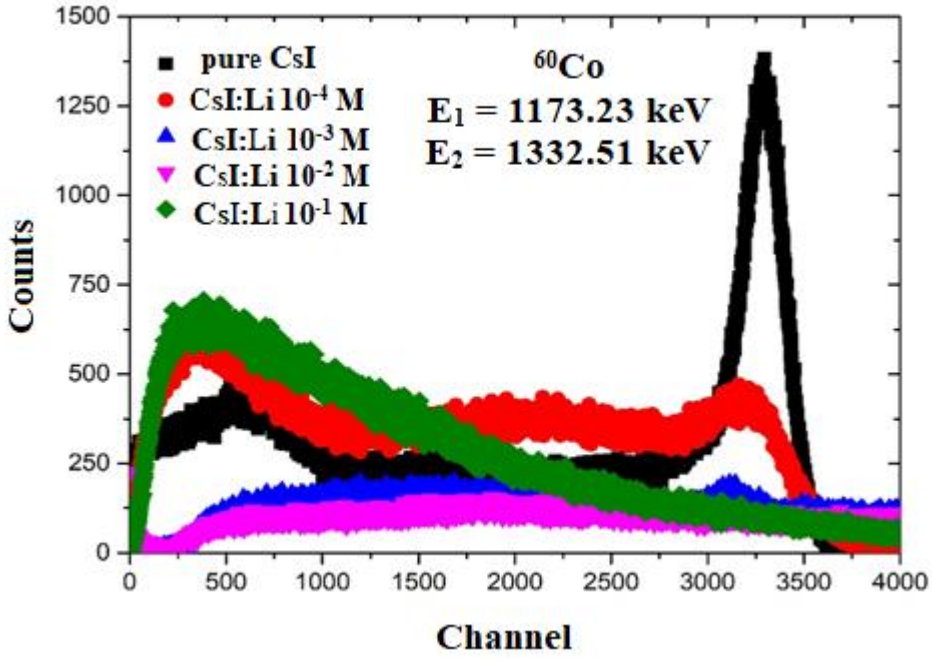


Figure 11 : Detection spectra obtained with CsI:Li and pure CsI crystals excited with gamma radiation from a ^{60}Co source.

The addition of lithium dopant in CsI crystals significantly influenced its behavior as a gamma detector. In Fig. 7 to 11 it can be seen that pulse height is dependent on Li

concentration.

The spectra shown in Fig. 7 to 11, for comparison of results, were all obtained under the same amplification and high voltage conditions. It can be noted that as the energy of the gamma emitting source increases, the spectra shift to channels that correspond to higher energies. therefore detection with these detectors has the characteristic of proportional detector.

In Fig. 7 only 1500 channels were presented, since the source energy is low and the spectrum is present in this region. It can be noted that the energy efficiency of CsI:Li 10^{-2} M and CsI:Li 10^{-3} M crystals is higher, as the responses of these detectors shift to the highest energy position.

In Fig.9, 10, 11 the response of the CsI:Li 10^{-2} M and CsI:Li 10^{-3} M crystals was not observed, because the spectra obtained with these two crystals for the operating condition saturated the measure system, getting off the spectrum in the multichannel. It can be noted that the resolution of these detectors is low, which is why they are not used for spectrometry.

The study of neutron radiation excited crystals performance was performed with an AmBe source that has energy from 1MeV to 12 MeV. The spectra were obtained individually with the neutron emitting source positioned 7 mm from the crystals. The Fig. 12 shows spectra obtained in the detection of neutron radiation.

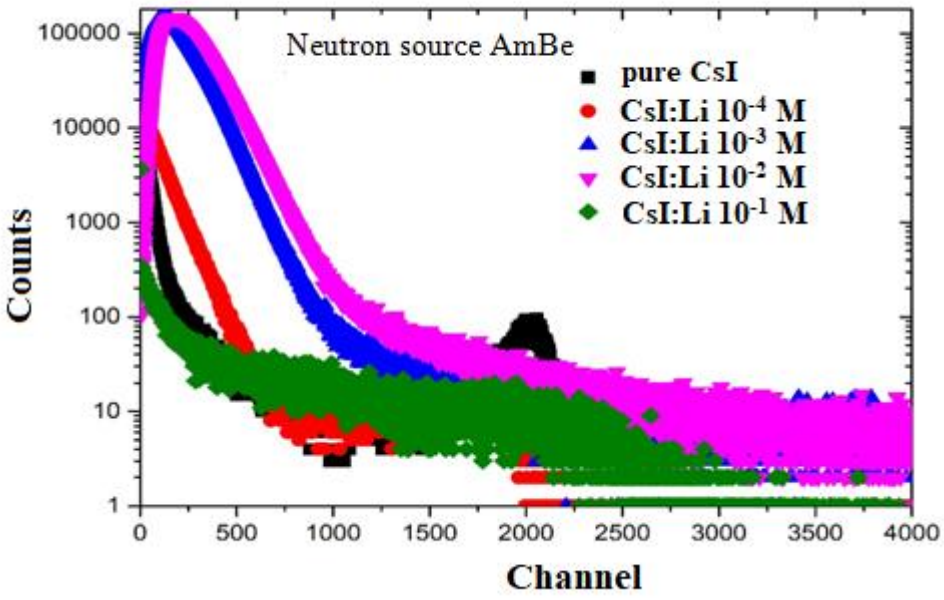


Figure 12 : Detection spectra obtained with CsI:Li and pure CsI crystals excited with neutron radiation from a AmBe source.

Figure 12 shows the spectra response of CsI:Li crystals with 10^{-4} M to 10^{-1} M concentrations and pure crystal when excited with neutrons radiation. Pure CsI crystal responded with a significant number of counts and a tendency to peak between the 1800 and 2200 channels. CsI:Li 10^{-4} M crystal, with the lowest dopant concentration, exhibited a very similar behavior resembling pure CsI crystal.

The CsI:Li crystals 10^{-2} M and 10^{-3} M showed detection spectra with a higher volume of counts than the other crystals, as opposed to comparisons with gamma radioactive sources. The significant detection improvement coul be attributed to the ability of the lithium doping element to interact with neutrons, justified by its high absorption shock section (640 barns). This demonstrates that lithium doped CsI crystals can be exploited in mixed-field neutron detection arrangements as they will not suffer significant gamma radiation interference and can provide more reliable neutron detection. [14]

The CsI:Li10^{-1} M crystal has the smallest volume of counts, with little response to interaction with neutrons even though it has the largest amount of lithium doping element. The lower behavior in neutron detection performance can be attributed to crystal lattice saturation, due to the amount of dopant.

The sensitivity of crystals to neutrons was verified using natural Li, which contains only a small fraction of the ^{6}Li isotope (7.5%) for the production of the investigated materials. This can be considerably improved by using enriched ^{6}Li.[15]

4. CONCLUSIONS

The vertical Bridgman technique used in this work was suitable for the growth of lithium doped CsI crystals.

The grown crystals showed appropriate optical qualities, showing a decrease in the transmittance value as the doping element concentration increases. The higher concentration CsI: Li crystal, 10^{-1} M, exhibited a significant decrease in transmittance when compared to pure CsI crystal.

The maximum emission peak luminescence spectra at 420 nm show good overlap with the quantum efficiency spectrum of the two alkaline photomultipliers, demonstrating the feasibility of using CsI: Li crystals as radiation detectors.

The addition of Li to the CsI matrix resulted in promising crystals when excited with gamma radiation in the energy range from 59 keV to 1333 keV, and showed characteristic of proportional detector.

The crystals showed sensitivity to neutron radiation, being that the CsI:Li crystals 10^{-3} M and 10^{-2} M showed better detection radiation in the concentration range of the studied Li dopant.

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