

## Preparation and Characterization of Thermal Neutron-Sensitive Lithium-Doped Polystyrene Plastic Scintillator

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### Abstract

Lithium-doped plastic scintillators enable dual-mode detection of fast neutrons and thermal neutrons, while retaining the advantages of conventional plastic scintillators such as low cost and large-volume fabrication capability. They have attracted considerable attention in recent years and show promising application prospects in thermal neutron detection. In this study, lithium methacrylate (Lithium Methacrylate, LiME)-doped polystyrene (Polystyrene, PS) plastic scintillators were successfully fabricated, and their properties were characterized. Through investigating the correlation between the radioluminescence intensity of the plastic scintillators and the mass concentrations of the initiator 2,2'-azobis(2-methylpropionitrile) (2,2'-Azobis(2-methylpropionitrile), AIBN), the primary fluor 2,5-diphenyloxazole (2,5-Diphenyloxazole, PPO), and the wavelength shifter 1,4-bis(5-phenyl-2-oxazolyl)benzene (1,4-Bis(5-phenyl-2-oxazolyl)benzene, POPOP), the optimal concentrations of AIBN, PPO, and POPOP in the plastic scintillator matrix were experimentally determined to be 0.1%, 2%, and 0.02%, respectively; through thermal polymerization, a series of highly transparent plastic scintillator samples with dimensions of 33 mm × 6 mm and LiME doping concentrations of 0–12% were successfully prepared, and the optical transmittance, photoluminescence, radioluminescence, and gamma energy spectrum response of these samples were characterized. The results indicate that the prepared lithium-doped plastic scintillators exhibit good optical quality, and the emission peak position shows limited shift compared with pure plastic scintillators. However, both the fluorescence intensity and relative light output decrease with increasing lithium doping concentration, which is speculated to be associated with fluorescence quenching effects induced by the doping process.

Full Text

Preamble

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**Preparation and Characterization of Thermal Neutron Sensitive Lithium-Doped Polystyrene Plastic Scintillators**

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**Abstract**

[Background] Lithium-doped plastic scintillators have attracted considerable attention in recent years for thermal neutron detection applications due to their dual-mode detection capability for both fast and thermal neutrons, combined with the advantages of conventional plastic scintillators such as low cost and scalability to large volumes. [Purpose] This study reports the successful preparation and characterization of polystyrene (PS) plastic scintillators doped with lithium methacrylate (LiME). [Methods] Through systematic investigation of the correlation between radioluminescence intensity and the mass concentrations of the initiator 2,2'-azobis(2-methylpropionitrile) (AIBN), primary fluorescent dye 2,5-diphenyloxazole (PPO), and wavelength shifter 1,4-bis(5-phenyl-2-oxazolyl)benzene (POPOP), the optimal concentrations were experimentally determined to be 0.1%, 2%, and 0.02%, respectively. Using a thermal polymerization method, a series of highly transparent plastic scintillator samples measuring 33 mm × 6 mm with LiME doping levels ranging from 0 to 12% were successfully fabricated. The optical transmittance, photoluminescence, radioluminescence, and gamma-ray spectral response of these samples were systematically characterized. [Results] The prepared lithium-doped plastic scintillators exhibited excellent optical quality, with limited shift in emission peak position compared to pure plastic scintillators. However, both fluorescence intensity and relative light output decreased with increasing lithium doping concentration, presumably due to fluorescence quenching effects induced by the doping process. [Conclusions] Large-volume lithium-doped plastic scintillators with high optical quality were successfully prepared. Although performance improvements are still needed, these scintillators show promising potential for applications requiring efficient dual-mode detection of fast and thermal neutrons.

**Keywords:** Thermal neutron detection, Lithium doping, Plastic scintillator, Preparation, Characterization

## Introduction

Thermal neutron detection plays a critical role in homeland security, weapons verification, nuclear physics, and medical imaging, driving continuous efforts to develop efficient thermal neutron detection materials. Historically,  $^3\text{He}$  gas detectors have been the preferred choice for thermal neutron detection, but they suffer from supply shortages, high costs, and the need for high-pressure processing. Consequently, low-cost, high-efficiency scintillation-based materials represent a major research direction as alternatives to  $^3\text{He}$ . Among existing thermal neutron scintillator materials,  $^6\text{LiF}/\text{ZnS}:\text{Ag}$  powders, lithium glass, and  $\text{Cs}_2\text{LiYCl}_6:\text{Ce}$  (CLYC) crystals are prominent examples, yet each faces limitations:  $^6\text{LiF}/\text{ZnS}:\text{Ag}$  powders exhibit poor transparency and low thermal neutron detection efficiency; lithium glass contains numerous internal defects with significant self-absorption of emitted light; and CLYC is prone to oxidation and moisture degradation with high manufacturing costs. Plastic scintillators offer advantages including short decay times ( $10^{-8}$ - $10^{-9}$  s), low cost, and the capability for large-volume fabrication, making them widely applicable. However, they show negligible response to thermal neutrons. Recently, doping plastic scintillators with elements having large thermal neutron capture cross-sections (such as  $^6\text{Li}$ ,  $^{10}\text{B}$ ,  $^{113}\text{Cd}$ , and  $^{157}\text{Gd}$ ) has attracted significant interest in the thermal neutron detection community, as this approach enables multi-mode detection of fast neutrons, thermal neutrons, and gamma rays.

In 2002, Normand et al. developed a boron-loaded plastic scintillator containing 1.5% p-terphenyl (PPP) and 0.01% POPOP, demonstrating its pulse shape discrimination (PSD) capability. In 2014, Pawelczak et al. prepared transparent boron-doped plastic scintillators by dissolving meta-carborane in styrene and methylstyrene matrices.  $^6\text{Li}$  offers several desirable characteristics as a neutron target material, including a reasonable capture cross-section, relatively high energy release upon neutron capture, and absence of gamma rays among the reaction products, making  $^6\text{Li}$  doping advantageous over  $^{10}\text{B}$ . However, research on  $^6\text{Li}$ -doped organic scintillators remains limited and progress has been slow, primarily due to the difficulty in preparing  $^6\text{Li}$ -doped plastic scintillators. The fundamental challenge lies in the low solubility of highly polar  $^6\text{Li}$ -containing compounds in nonpolar aromatic organic scintillator matrices. Consequently, most reported materials are organic-inorganic composite scintillators, where nano- or micro-scale  $^6\text{Li}$ -containing inorganic compound particles are physically dispersed in a plastic matrix via ultrasonication. For example, Rajakrishna et al. dispersed LiF powder in plastic scintillators, achieving approximately 30% improvement in neutron sensitivity, but such composite scintillators suffer from non-uniform powder dispersion and poor optical quality, limiting practical applications.

To address these challenges, this study introduces hydrocarbon chains to increase the probability of forming stable nonpolar solvent shells, combining lithium-containing compounds with coordinating solvents to form low-polarity molecular complexes. These complexes effectively increase LiME doping levels

through copolymerization with a polar comonomer—methacrylic acid (MAA) —added to the plastic scintillator matrix. Using thermal polymerization, we successfully fabricated a series of large, highly transparent doped plastic scintillators and investigated the effects of LiME doping concentration on their optical and scintillation performance.

## Experimental Methods

### 1.1.1 Raw Materials

The raw materials used included styrene monomer (SM, 99.5% purity, Aladdin), methacrylic acid (MAA, 99% purity, Aladdin), 2,2'-azobis(2-methylpropionitrile) (AIBN, 95% purity, Aladdin), lithium hydroxide (LiOH, 98% purity, Aladdin), methanol (CH<sub>3</sub>OH, 99.5% purity, Aladdin), acetone (AC, 99.5% purity, Sigma-Aldrich), 2,5-diphenyloxazole (PPO, 99% purity, Aladdin), 1,4-bis(5-phenyl-2-oxazolyl)benzene (POPOP, 99% purity, Aladdin), and sodium chloride (NaCl, 99.5% purity, Aladdin). Commercially available SM typically contains polymerization inhibitors such as tert-butylcatechol and other impurities that can cause spontaneous polymerization during storage or transport; therefore, purification by vacuum distillation was performed before use. MAA was dried with NaCl and purified by vacuum distillation to remove inhibitors. All other chemicals were used as received without further treatment.

### 1.1.2 Preparation of Plastic Scintillators

SM, PPO, POPOP, and AIBN were added to a glass vial, which was then sealed and evacuated before backfilling with argon to prevent oxygen incorporation during ultrasonication, which could cause fluorescence quenching in the plastic scintillator. The vial was placed in an ultrasonic cleaner and sonicated for 30 minutes to ensure complete dissolution of PPO, POPOP, and AIBN in SM. The vial was then transferred to an electric drying oven and heated at a rate of 5 °C · h<sup>-1</sup> to 50 °C, where it was held for 24 hours to allow pre-polymerization. Subsequently, the temperature was increased to 80 °C and maintained for 3 days to ensure complete polymerization of the SM matrix. After cooling to room temperature, the samples were cut and polished to obtain plastic scintillators with various compositions.

### 1.1.3 Synthesis of LiME

LiOH was dissolved in a measured amount of CH<sub>3</sub>OH solution, and excess MAA was added. The mixture was stirred for 3 hours to ensure complete reaction, then poured into excess AC solution to precipitate LiME. The precipitate was filtered and redissolved in CH<sub>3</sub>OH, followed by reprecipitation with AC. This purification cycle was repeated multiple times. The final product was dried in a vacuum oven at 60 °C to obtain LiME powder.

### 1.1.4 Preparation of LiME-Doped Polystyrene Plastic Scintillators

The synthesis schematic for Li-doped plastic scintillators and photographs of the prepared samples are shown in Figure 1: see original paper and (b). Different masses of LiME along with predetermined amounts of PPO, POPOP, and AIBN were dissolved separately in glass vials containing MAA and SM, each sonicated for 5 minutes to ensure homogeneous mixing. The MAA solution was then added to the SM-containing vial, which was sealed, evacuated, and backfilled with argon. After sonicating for 30 minutes to achieve uniform mixing, the vial was placed in an oven and heated at  $5\text{ }^{\circ}\text{C}\cdot\text{h}^{-1}$  to  $50\text{ }^{\circ}\text{C}$  for pre-polymerization for 24 hours, then ramped at  $5\text{ }^{\circ}\text{C}\cdot\text{h}^{-1}$  to  $80\text{ }^{\circ}\text{C}$  and held for 3 hours, before cooling to room temperature at  $5\text{ }^{\circ}\text{C}\cdot\text{h}^{-1}$ . Plastic scintillators with various LiME doping concentrations were prepared, and all samples were polished into cylinders with a diameter of 33 mm and thickness of 6 mm.

### 1.2 Performance Testing and Characterization

The composition and structure of LiME were analyzed using a PerkinElmer Spotlight 400&Frontier Fourier-transform infrared spectrometer and a Renishaw inVia variable-temperature Raman spectrometer. Optical transmittance of the plastic scintillators was measured using a PerkinElmer Lambda 950 UV-Vis-NIR spectrophotometer over a wavelength range of 200–800 nm. Photoluminescence (PL) properties were characterized using an Edinburgh Instruments FLS920 fluorescence spectrometer with a xenon lamp (Xe) as the excitation source. X-ray excited luminescence (XEL) spectra were obtained using an Amptek Mini-X-OEM X-ray source as the excitation source, with the measurement setup shown schematically in [Figure 2: see original paper]. In this configuration, fluorescence emitted from the sample under X-ray excitation was collected by a lens and directed into an emission monochromator. Pulse height spectra (PHS) were acquired using a custom-built gamma spectrometer with a  $^{137}\text{Cs}$  source (662 keV) as the excitation source and a Hamamatsu R2059 photomultiplier tube (PMT). The measurements were conducted at a bias voltage of  $-1800\text{ V}$ , gain of 45, and shaping time of 2 s.

## Results and Discussion

### 2.1.1 Effect of PPO Concentration on Scintillator Performance

PPO serves as the fluorescent dye in plastic scintillators, absorbing radiation energy and converting it into visible light. To investigate the relationship between scintillator performance and PPO concentration, plastic scintillators containing various PPO mass concentrations (1.0%, 1.5%, 2.0%, 2.5%, and 3.0%) were prepared while maintaining POPOP and AIBN concentrations at 0.02% and 0.1%, respectively. The XEL spectra and integrated XEL intensity as a function of PPO concentration are shown in [Figure 3: see original paper]. At low concentrations ( $< 2.0\%$ ), the XEL intensity increased with PPO concentration. However, further increases in PPO concentration led to a turning point where

the XEL intensity began to decrease, suggesting that strong intermolecular interactions at higher concentrations cause concentration quenching. This effect increases the probability of non-radiative transitions for excited electrons, causing more radiation energy to be converted into vibrational energy of  $\pi$ -electrons and dissipated as heat, thereby reducing XEL intensity.

### 2.1.2 Effect of POPOP Concentration on Scintillator Performance

During the scintillation process,  $\pi$ -electrons undergo de-excitation through non-radiative pathways. However, the limited amount of radiation energy dissipated as heat results in partial overlap between the short-wavelength emission spectrum and the long-wavelength absorption spectrum, leading to small Stokes shifts and severe self-absorption effects that hinder light transmission within the scintillator. Adding a wavelength shifter such as POPOP increases the Stokes shift, significantly reducing self-absorption during light propagation and shifting the emission band of the PPO-doped scintillator into the sensitive wavelength range of the PMT, thereby improving detection efficiency. Nevertheless, its introduction also affects scintillator performance to some extent. To investigate the relationship between POPOP concentration and scintillator performance, plastic scintillators with POPOP concentrations of 0.01%, 0.015%, 0.02%, 0.025%, 0.03%, and 0.04% were prepared while maintaining PPO and AIBN concentrations at 2.0% and 0.1%, respectively. The XEL spectra and integrated intensity as a function of POPOP concentration are presented in [Figure 4: see original paper].

As shown in [Figure 4: see original paper], the XEL intensity initially increased with POPOP concentration, reaching a maximum at 0.02% POPOP. Further increases in POPOP concentration ( $> 0.02\%$ ) led to concentration quenching and reduced XEL intensity. In plastic scintillators, POPOP content is typically very low (0.001–0.1%), so although increasing its concentration beyond the optimum continues to affect performance, the impact is relatively small and gradually stabilizes.

### 2.1.3 Effect of AIBN Concentration on Scintillator Performance

Initiators generally accelerate the polymerization process and reduce reaction time. To improve the efficiency of large-scale scintillator fabrication, the effect of AIBN concentration on scintillator performance was investigated. Plastic scintillators with varying AIBN concentrations (0.05%, 0.1%, 0.2%, and 0.3%) were prepared while maintaining the fluorescent dye combination of 2.0% PPO and 0.02% POPOP. The XEL spectra and integrated intensity as a function of AIBN concentration are shown in [Figure 5: see original paper].

The XEL intensity decreased progressively with increasing AIBN concentration. During polymerization, AIBN may act as an impurity in the plastic scintillator, creating fluorescence quenching centers that reduce scintillation efficiency. Considering both polymerization time and luminescence performance, an AIBN

concentration of 0.1% was selected for subsequent preparations, as this provides a good balance between high luminescence intensity and reasonable polymerization time, avoiding prolonged high-temperature polymerization that could cause discoloration and macroscopic defects.

In summary, through systematic investigation of component concentration effects, the optimized matrix composition was determined to be 2.0% PPO as the primary fluorescent dye, 0.02% POPOP as the wavelength shifter, and 0.1% AIBN as the initiator.

## 2.2 Composition and Structure Characterization of LiME

To confirm successful synthesis of LiME, Fourier-transform infrared spectroscopy (FTIR) and Raman spectroscopy (RS) were employed to characterize its composition and structure, with results shown in Figure 6: see original paper and (b), respectively. The FTIR spectrum shows absorption peaks at 2970  $\text{cm}^{-1}$  and 2936  $\text{cm}^{-1}$ , corresponding to asymmetric C-H stretching vibrations in  $\text{CH}_3$  and  $\text{CH}_2$  groups, respectively. The peak at 1652  $\text{cm}^{-1}$  is attributed to C=C stretching vibration, while peaks at 1570  $\text{cm}^{-1}$  and 1414  $\text{cm}^{-1}$  correspond to asymmetric and symmetric stretching vibrations of carboxylate groups. In MAA, a broad O-H absorption peak appears around 2976  $\text{cm}^{-1}$ , but this peak is absent in the LiME FTIR spectrum, indicating that the hydrogen on the -OH group has been replaced by lithium. The FTIR spectrum matches reported data for LiME in the literature. The Raman spectrum of LiME at room temperature under non-polarized, micro-confocal conditions is shown in Figure 6: see original paper. The peak at 3101  $\text{cm}^{-1}$  corresponds to =C-H stretching vibrations in unsaturated bonds, while peaks at 2990  $\text{cm}^{-1}$  and 2932  $\text{cm}^{-1}$  are assigned to asymmetric and symmetric C-H stretching vibrations, respectively. The peak at 1648  $\text{cm}^{-1}$  is attributed to C=O stretching in -COO groups, the 1538  $\text{cm}^{-1}$  peak to C=C stretching, and the 1428  $\text{cm}^{-1}$  peak to -COO stretching. Since LiME is an ionic compound, metal-ion bonds do not appear in the Raman spectrum. These combined results confirm successful synthesis of LiME.

## 2.3 LiME-Doped Plastic Scintillators

LiME was incorporated into the SM matrix via thermal polymerization to prepare lithium-doped plastic scintillators with doping levels up to 12% and good optical quality. After cutting and polishing, cylindrical samples with a diameter of 33 mm and thickness of 6 mm were obtained. [Figure 7: see original paper] shows photographs of the large-size plastic scintillator samples with LiME doping concentrations ranging from 0 to 12%, corresponding to lithium doping levels of 0 to 0.905%. The samples exhibited a gradual color change from colorless to yellow with increasing LiME content, with the cause of this discoloration requiring further investigation.

The optical transmittance of the samples is shown in Figure 8: see original paper.

The dashed line represents the XEL spectrum of the LiME-doped plastic scintillators, with the emission peak maximum at 420 nm. The emission-weighted light transmittance (EWLT) is defined as:

$$EWLT = \frac{\int LT(\lambda) \cdot Em(\lambda) d\lambda}{\int Em(\lambda) d\lambda}$$

where  $LT(\lambda)$  and  $Em(\lambda)$  are the optical transmittance and fluorescence intensity at wavelength  $\lambda$ , respectively. Compared to transmittance at the emission peak alone, EWLT provides a more accurate reflection of scintillation light transmission capability. Figure 8: see original paper shows that EWLT values decrease upon LiME doping, but all samples maintain EWLT above 51%, indicating reasonably good optical quality. The sample with 1% LiME showed anomalous behavior, likely due to poorer surface polishing quality.

To evaluate the luminescence performance of LiME-doped scintillators, XEL spectra were measured for samples with various doping levels, with integrated intensity comparisons shown in Figure 8: see original paper. The XEL emission maximum remained at approximately 420 nm, with no significant peak shift induced by LiME doping. The XEL intensity decreased sharply as LiME doping increased from 0% to 1%, with the 1% LiME sample showing only about 36% of the emission intensity of the pure plastic scintillator. Further increases in LiME doping from 1% to 12% resulted in a gradual, slow decline in XEL intensity, as illustrated in the inset of Figure 8: see original paper.

Photoluminescence excitation (PLE) and emission (PL) spectra for 0% and 10% LiME-doped plastic scintillators are presented in [Figure 9: see original paper]. Both samples show similar excitation and emission profiles, though with slight red shifts in the peak maxima. The emission peaks are located at 420 nm and 423 nm, while the excitation peaks shift from 296 nm to 302 nm. The shoulder at 362 nm originates from the primary scintillator PPO, while the peaks at 420 nm and the shoulder at 450 nm correspond to emission from the secondary fluor POPOP. Since photoluminescence was measured in surface mode without volume absorption effects, characteristic emission peaks from both PPO and POPOP were observed. LiME doping did not introduce new emission peaks, and fluorescence remained primarily from the dye molecules. The slight red shift in both excitation and emission peaks after LiME doping may be attributed to minor energy level structure adjustments caused by LiME-induced structural distortions or to absorption effects related to significant yellowing of the scintillator, requiring further investigation.

To investigate the correlation between LiME doping concentration and scintillator light output, pulse height spectra under  $^{137}\text{Cs}$  excitation were measured for pure and doped scintillators, as shown in [Figure 10: see original paper]. The cylindrical 33 mm  $\times$  6 mm samples were wrapped with polytetrafluoroethylene tape and optically coupled to a Hamamatsu R2059 PMT using Dow Corning DC-200 silicone oil. Due to the low photoelectric detection efficiency of plastic

scintillators for 662 keV gamma rays, quantitative comparison of light output using the photopeak is not feasible. In Compton scattering, gamma rays are scattered by free electrons, and when the scattered gamma ray escapes the detector, the electron's energy is deposited in the detector. The recoil electrons have a well-defined maximum energy at the Compton edge, corresponding to the maximum energy transfer from the gamma ray to the electron. The Compton edge value can be used for energy calibration and for quantitative comparison of relative light output (LO) from plastic scintillators. Pulse height spectra of pure and doped scintillators were measured using a  $^{137}\text{Cs}$  source, where the channel number corresponding to the Compton edge is proportional to the light output. Using this relationship, the relative light output of doped scintillators compared to pure scintillators was calculated, with results summarized in .

Both [Figure 10: see original paper] and demonstrate that the Compton edge shifts progressively to lower channel numbers with increasing LiME doping, indicating decreasing light output. Possible explanations include: (1) reduced optical transmittance after LiME doping, as shown in Figure 8: see original paper, leading to lower scintillation photon transmission; (2) LiME doping may affect the energy transfer process in plastic scintillators or dissipate absorbed energy through non-radiative pathways such as molecular vibrations, reducing emission intensity; and (3) MAA addition may influence energy transfer processes in the scintillator, such as interactions between heteroatoms (N, O) on PPO and polar acid functional groups on MAA, thereby reducing light output.

## Conclusion

Polystyrene-based plastic scintillators with various compositions were prepared via thermal polymerization. By systematically investigating the relationship between component concentrations and scintillation performance, the optimal concentrations of AIBN, PPO, and POPOP were determined to be 0.1%, 2%, and 0.02%, respectively. To enhance the probability of forming stable nonpolar solvent shells through hydrocarbon chains, lithium-containing compounds were combined with coordinating solvents to form low-polarity molecular complexes. Through copolymerization with the polar comonomer MAA added to the scintillator matrix, LiME doping levels were effectively increased, enabling successful fabrication of highly transparent plastic scintillators with LiME doping up to 12% (equivalent to 0.905% Li doping). Characterization of scintillators with various LiME doping levels revealed that LiME doping had limited effect on the emission spectral shape but caused reductions in radioluminescence intensity and light output (Compton edge). The prepared lithium-doped polystyrene plastic scintillators offer advantages of low cost, simple fabrication, and high transparency, showing good potential for dual-mode fast/thermal neutron detection and large-area radiation imaging applications. Due to time and scope limitations, this study focused on preparation and basic optical/scintillation characterization. Future work should optimize fabrication parameters to improve optical quality and scintillation performance, utilize  $^6\text{Li}$ -enriched organic

lithium salts instead of natural-abundance salts to enhance thermal neutron detection efficiency, and conduct comprehensive thermal neutron detection performance evaluations to advance practical applications of lithium-doped plastic scintillators.

## Author Contributions

Kaihui Zhang conducted the experiments, performed data analysis, and drafted the manuscript. Xiang Li designed the overall experimental scheme, evaluated results, and revised the manuscript. Mingxue Deng and Yun Shi contributed to result discussion and manuscript revision. Qi Zhou assisted with data analysis. Ke Wang assisted with manuscript framework design and provided guidance for revision. Junfeng Chen designed the overall manuscript framework, supervised the study, and secured research funding.

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