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An organosilicon-loaded plastic scintillator synthesized for neutron/gamma discrimination

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Abstract

In this work, we report a scintillator used for neutron/gamma discrimination, which consists of a polyvinyltoluene matrix loaded with monomers of dimethoxydiphenylsilane (DDS), and fluor dyes, namely 2,5-diphenyloxazole and 7-diethylamino-4-methylcoumarin as the primary fluorophore and wavelength-shifting, respectively. The DDS is an organosilicon compound containing double benzene rings that facilitates fluorescence emission and radiation resistance. Several measurements were performed to explore the scintillators optical and detection properties, including 238Pu-Be neutron radiation and relative light yield conducted by 137Cs gamma-ray source. It was found that the synthesized plastic scintillators were capable of excellently discriminating between neutron and gamma-rays. They exhibited a light output that were comparable to the commercial scintillator (EJ-200). The scintillators are low-cost productive, highly transparent, and somewhat soft but hard enough for post-production processing (machine cutting and polishing). This design may contribute a new strategy for highly efficient neutron/gamma discrimination organic scintillators.

Full Text

Preamble

An organosilicon-loaded plastic scintillator synthesized for neutron/gamma discrimination

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We report a novel scintillator for neutron/gamma discrimination consisting of a polyvinyltoluene matrix loaded with dimethoxydiphenylsilane (DDS) monomers and fluor dyes—2,5-diphenyloxazole as the primary fluorophore and 7-diethylamino-4-methylcoumarin as the wavelength shifter. DDS is an organosilicon compound containing double benzene rings that facilitates fluorescence emission and enhances radiation resistance. We performed comprehensive measurements to characterize the scintillators' optical and detection properties, including ^{238}Pu-Be neutron irradiation and relative light yield measurements using a ^{137}Cs gamma-ray source. The synthesized plastic scintillators demonstrated excellent neutron/gamma discrimination capability with light output comparable to the commercial scintillator EJ-200. These scintillators are cost-effective to produce, highly transparent, and exhibit moderate softness while remaining sufficiently rigid for post-production processing (machining and polishing). This design offers a new strategy for developing highly efficient organic scintillators for neutron/gamma discrimination.

Keywords: Neutron detection, Plastic scintillation, Pulse shape discrimination, Polymethyl-methacrylate, Polystyrene

INTRODUCTION

Neutron detection based on organic scintillators has been widely employed in nuclear and particle physics experiments for many decades, particularly in high-radiation environments for applications such as beam luminosity measurement, particle tracking, and time-of-flight particle separation [1, 2]. Such measurements are invariably accompanied by background gamma rays from neutron activation of surrounding materials, and organic scintillators are inherently sensitive to gamma rays due to their organic composition. Consequently, the ability to identify different types of incident particles—known as pulse shape discrimination (PSD) [3, 4]—is essential for organic scintillator-based neutron spectroscopy when detecting high-energy neutrons in the presence of gamma rays.

For many years, neutron detection has predominantly relied on ^{3}He proportional counters due to their high detection efficiency for thermal neutrons [5–7]. However, the extremely high cost of ^{3}He has limited its widespread use and has stimulated intensive research into alternative, less expensive materials. Among PSD-capable organic scintillators, robust and inexpensive plastic scintillators [8, 9] have attracted considerable interest for large-scale scientific facility construction, border monitoring, and reactor surveillance applications, especially when compared with single-crystal and liquid scintillators that face challenges related to high cost, transportation of large-area detectors, and safety concerns (toxicity, flammability) in field operations [10].

The most commonly used plastic scintillators are tertiary mixtures of a polystyrene (PS) or polyvinyltoluene (PVT) base with small amounts of

primary fluorophore and secondary wavelength-shifter dyes [11]. Since the base material' s intrinsic light yield is low due to self-absorption, both the selection of the two dye types and their concentrations are crucial for establishing an efficient network for excitation energy migration and triplet annihilation that enables PSD [12, 13]. PS and PVT are aromatic hydrocarbon polymers containing benzene rings bonded to methyl (CH_3-) and vinyl (CH_2-CH-) groups. When irradiated with high-energy particles, the molecular chains in these scintillators suffer damage, breakage, and cross-linking [1], leading to changes in polymer chemical structure and formation of free radicals (color centers), followed by yellowing and deterioration of light yield, transmittance, and mechanical properties [14, 15].

The radiation resistance of organic scintillators can be attributed to the bond dissociation energies of their backbone atoms (e.g., 3.59 eV for C–C, 3.71 eV for C–O, and 4.61 eV for Si–O bonds) [15]. Polysiloxane, an organosilicon polymer featuring a backbone of alternating Si–O–Si bonds with organic substituent groups, is characterized by high bond energy and excellent radiation resistance. Studies have demonstrated that polysiloxane-based scintillators show no yellowing after radiation doses up to 100 kGy [16] and can discriminate neutrons in gamma-ray backgrounds [17]. However, they are inherently soft due to their elastic nature, and their PSD performance remains inferior to conventional PS- and PVT-based scintillators despite optical property improvements achieved through cross-linking strategies [18].

In this work, we developed a novel PSD organic scintillator that combines the advantages of both plastic and polysiloxane-based scintillators by employing 2,4-diphenyl-1,3,2-dioxasilolane (DDS) with PVT resin as the matrix and incorporating 2,5-diphenyl-oxazole (PPO) and 7-diethylamino-4-methylcoumarin (MDAC) as primary fluorophore and wavelength shifter, respectively. The DDS monomers polymerized into linear segments that were inserted into the PVT cross-linked network through low-density polymerization, yielding bulk scintillators with some elasticity while remaining sufficiently rigid for mechanical processing. The synthesized scintillators are cost-effective, transparent, and exhibit outstanding PSD capability, making them promising candidates for commercial applications. Furthermore, their light yields are comparable to commercially available plastic scintillators (EJ-200).

II. SAMPLES FABRICATION AND CHARACTERIZATION

Vinyltoluene monomers (C_9H_{10}, >98%, m- and p-mixture, containing TBC stabilizer) were purchased from TCI Chemicals (Shanghai) Industrial Development Co., Ltd. DDS, MDAC, and the initiator 2,2' -Azobis(2-methylpropionitrile) (AIBN, >98%) were obtained from Shanghai Macklin Biochemical Co., Ltd. PPO was purchased from Shanghai YuanYe Biotechnology Co., Ltd., and divinylbenzene (DVB, containing stabilizer) was obtained from Meryer (Shanghai) Chemical Technology Co., Ltd.

Table 1 . DDS monomer concentration in mass weight percent (wt%) relative to the total base, and Shore hardness (unit: HD) measured using a portable digital durometer (Shore A hardness meter). Five equidistant points on a flat side of each sample were measured and averaged to obtain the hardness value.

Samples	DDS (wt%)	Shore hardness (HD)
RK1	20	45.5 ± 0.8
RK2	25	41.4 ± 0.5
RK3	30	39.7 ± 0.3
RK4	35	39.3 ± 0.9
RK5	40	37.2 ± 0.5

The synthesis procedure followed our previous work [11]. Prior to sample preparation, vinyltoluene monomers and divinylbenzene were passed through an aluminum oxide column to remove stabilizers, then mixed and stirred at room temperature for three minutes. The mixture was sonicated until homogeneous and clear. Subsequently, fluorophores (30 wt% PPO, 0.2 wt% MDAC), radical polymerization initiator (0.05 wt% AIBN), and cross-linking agent (2 wt% DVB) were added to the mixture, stirred for an additional 2–5 minutes, and transferred to flat-bottom glass vials for ultrasonication until complete dissolution of dopants. The mixed solution was poured into glass vials and subjected to at least three freeze-pump-thaw cycles using liquid nitrogen for rapid freezing, vacuum degassing (3–5 min), and thawing at room temperature to completely remove dissolved oxygen. The vials were sealed under oxygen-free atmosphere and heated in a thermostat to promote radical thermal polymerization. To prevent explosive polymerization, the temperature was maintained at 50.0°C for two days, then slowly increased from 80.0°C to 110.0°C at 3°C/h and held for 24 h. To prevent crack and bubble formation, the polymers were slowly cooled to room temperature at 3°C/h during annealing, then placed in a constant-temperature environment at 50.0–60.0°C for internal stress release. Scintillator samples with varying DDS amounts (Table 1) measuring approximately 20.0 mm in diameter and 10.0 mm in thickness were obtained by carefully breaking the vials after cooling to room temperature.

The scintillators were cut and polished to match the dimensions of a control EJ-200 sample, measured using a Mitutoyo micrometer (Mitutoyo Corp.), with thickness differences not exceeding 100 µm. Shore hardness was evaluated using a portable digital durometer (Shore A hardness meter, Taizhou AI Measurement Instrument Co., Ltd.), with five equidistant points measured on a flat side of each sample and averaged. Elastic recovery testing and excitation-emission spectra were performed using a metallographic microscope (BX53M, Olympus Corporation) and an FLS1000 fluorescence spectrometer (Edinburgh Instruments), respectively. Ultraviolet-visible transmission spectra were recorded using a UV-Vis spectrophotometer (PerkinElmer Lambda 750) over a wavelength range of 250–800 nm. Thermal decomposition behavior was studied from 30°C to 800°C

using a thermogravimetric analyzer (STA 449 F3 Jupiter, Netzsch GmbH, Germany). Fourier-transform infrared spectra (FTIR) were obtained using a Nicolet 6700 spectrometer (Thermo Fisher Scientific, USA) to analyze structural and functional group information.

PSD performance was studied by irradiating the scintillators with a 20 Ci ^{238}Pu-Be neutron source at the University of South China, employing an 8-channel 14-bit 500 MS/s flash ADC waveform digitizer (CAEN DT5730). All samples were wrapped in three layers of polytetrafluoroethylene (PTFE) tape on all surfaces except the one coupled to a Hamamatsu H1949-51 PMT using optical-grade dimethyl silicone (Dow Corning, PMX-200). Signals from the PMT anode were converted to digital waveforms and analyzed using CAEN DPP-PSD firmware, which combines digital QDC (charge integration) with shape discrimination for particle identification. A ^{60}Co source (1.17 MeV, 1.33 MeV) together with ^{137}Cs (662 keV) was used for electron-equivalent energy calibration by Gaussian fitting of the Compton edge positions. Light output (LO) was determined from the maximum position of the Compton edge in the ^{137}Cs pulse height spectra and compared with a three-year-old commercial EJ-200 sample to obtain relative LO. To assess stability, measurements were repeated three times at 2 days, 96 days, and 175 days after preparation. All experimental conditions—including digitizer settings, PMT operation at -1600 V (ISEG Cor), 10 cm source-to-sample distance, and lead brick shielding—were maintained constant to ensure consistency.

III. RESULTS AND DISCUSSION

A. Physical Properties

DDS is a cost-effective silane compound with double benzene rings and remarkable radiation resistance, capable of efficiently dissolving many organic dyes. The aromatic matrix (PVT/DDS) inherently emits fluorescence in the ultraviolet spectrum between 250 and 380 nm [19]. To minimize self-absorption, the PPO-MDAC dye pair was selected to shift the scintillator emission wavelength to 420–450 nm, which matches well with the spectral sensitivity of silicon photomultipliers and avalanche photodiodes. PPO is a commonly used primary fluorophore with high solubility in aromatic solvents, while MDAC serves as an efficient secondary dye with high light output for routine plastic scintillator preparation. The excellent PSD capability resulting from this dye pair can be understood through fundamental energy transfer principles [20].

With 20–40 wt% DDS, the synthesized scintillators remained colorless and optically transparent without yellowing or surface dye precipitation visible to the naked eye even after six months (Fig. 1 [Figure 1: see original paper]). However, when DDS content exceeded 40 wt%, microcrack formation and yellowing were observed (Fig. 2 Figure 2: see original paper-Top). Mechanical hardness decreased with increasing DDS concentration (Table 1). Since complete constituent analysis is nearly impossible for organic scintillators, we inferred

that no heterogeneous cross-linking occurred between DDS and vinyltoluene monomers, as FTIR analysis revealed only one additional characteristic absorption line at 1065.59 cm^{-1} corresponding to the Si–O–Si bond when comparing the 30 wt% DDS sample with pure PVT (0 wt% DDS) (Fig. 2(b)). During thermal polymerization, DDS monomers likely cross-linked among themselves to form low-molecular-weight chains that became embedded in the vinyltoluene cross-linked network, reducing bulk mechanical hardness.

This reduced hardness from the elastomeric DDS is not necessarily detrimental, particularly regarding elastic recovery. To verify this, a blunt needle (0.79 mm diameter) was pressed into a 30 wt% DDS sample for one minute, and surface morphology evolution was recorded by metallographic microscopy (Fig. 3 [Figure 3: see original paper]). The cylindrical indentation reached 1.5 mm depth but restored to a nearly flat plane within 30 seconds and completely disappeared after 60 seconds, with no significant extrusion traces, demonstrating that DDS addition imparts beneficial elasticity. This behavior arises from high mobility of the siloxane main chains due to the low energy barrier for bending and torsion of Si–O–Si linkages [21].

Thermal stability was assessed by thermogravimetric analysis (TGA) under nitrogen atmosphere from 30°C to 800°C at 10°C/min (Fig. 2(c)). No mass loss occurred initially; weight loss began above 120°C with similar thermogravimetric profiles for all samples, corresponding to inter- and intrachain reactions. Decomposition products included volatile linear oligomers and fluorophores (PPO, MDAC). Compared with the 0 wt% DDS scintillator (decomposition temperature ~160°C), the DDS-loaded samples showed decreased initial decomposition temperature, indicating improved flexibility from embedded DDS low-molecular-weight chains. Above 210°C, the thermal degradation rate showed slight, complex dependence on DDS concentration that warrants further investigation. In the second stage (260–380°C), DDS samples exhibited greater weight loss than pure PVT due to short-chain DDS degradation, indicating reduced stability in this range. In the third stage (400–800°C), samples continued degrading, leaving approximately 2–5% solid residue, primarily SiOC carbonaceous material after mineralization [22].

B. Optical Performance

The synthesized scintillators emit fluorescence in the 400–600 nm range with a characteristic peak at ~434 nm in the excitation-emission spectra (Fig. 2(d)), indicating efficient singlet excitation energy transfer from PPO to MDAC molecules. The DDS samples exhibit high transparency (Fig. 2(e)), which is primarily limited by matrix self-absorption and dye aggregation [19]. At low DDS loading, monomers were sparsely dispersed and many did not participate in polymerization, acting as photon scattering centers. As DDS increased from 20 wt% to 30 wt%, transparency improved due to consumption of monomers in polymerization reactions forming linear segments. However, above ~35 wt% DDS, two competing effects reduced transparency: (1) high benzene

ring density introduced strong π-π stacking interactions that promoted dye aggregation of fluor molecules in the tertiary scintillator, and (2) substantial filling of the PVT cross-linked network by DDS linear chains generated shear stress and microcracks due to plasticity discrepancies between the two phases (Fig. 2(a)-Top).

Benzene rings with π-electrons enhance fluorescence emission. Replacement of PVT with DDS increased photoluminescence intensity (Fig. 2(f)), confirming that DDS double benzene rings positively contribute to scintillation emission. However, excessive DDS (>30 wt%) caused benzene ring stacking, creating physically compact proximity that rearranged electron energy levels by splitting ground and first excited states. Consequently, fluorescence intensity decreased with increasing DDS, likely due to aggregation-caused quenching (ACQ).

Additionally, DDS samples prepared with 50 wt% PPO remained transparent after 15 days of storage (Fig. 2(a)-Bottom), demonstrating that DDS incorporation enhances fluor molecule dissolution. Combined with our previous work [18], we hypothesize that when small amounts of monomeric substances participate in cross-linked network construction, they help prevent dye aggregation by isolating molecules within the subdivided network grid, thereby enhancing fluor solubility.

C. PSD and Relative Light Output

Samples were irradiated with the ^{238}Pu-Be source to evaluate PSD performance. The PSD principle relies on different proportions of prompt and delayed components in scintillation pulses from different particles. When an ionizing particle traverses the scintillator, deposited energy excites π-electrons to singlet and triplet states, which subsequently de-excite to the lowest singlet (S_1) and triplet (T_1) states. Fast fluorescence emission from $S_1 \rightarrow S_0$ transition (10^{-9}-10^{-8} s) provides the prompt component. Since direct $T_1 \rightarrow S_0$ transition is spin-forbidden, the delayed component arises from triplet–triplet annihilation ($T_1 + T_1 \rightarrow S_1 + S_0$) followed by slower phosphorescence emission ($S_1 \rightarrow$ h) over longer timescales (10^{-4} s). The T_1 state density depends on the ionizing power of incident particles, providing the basis for PSD. Heavy charged particles produce higher T_1 concentrations, yielding more pronounced slow components than light particles.

PSD capability was quantitatively evaluated using the charge integration method [23]. Integrating a short-time gate (Q , fast component) and a long-time gate (Q) on digital waveforms yields a PSD factor: PSD = 1 - Q /Q , where Q and Q represent integrations from waveform onset to the nearby peak and the entire waveform, respectively. Two-dimensional PSD vs. Q plots clearly separate neutrons (higher PSD values) from gamma rays (lower PSD values). The figure of merit (FOM) quantifies neutron/gamma discrimination: FOM = S/(FWHM + FWHM^n), where S is the distance between gamma and neutron peak positions, and FWHM and FWHM^n are the full widths at half-maximum of the respective peaks obtained by Gaussian

fitting.

Figure 4 [Figure 4: see original paper] and Fig. 5(a) show the 2D PSD distributions and relative LO for DDS samples two days after synthesis. The scintillators clearly separated neutrons from gamma rays. Silane-based LO is inherently less favorable than PVT-based LO, so relative LO decreased monotonically with PVT replacement by DDS (Fig. 5(a)). Although LO reduction typically degrades discrimination performance, the increased benzene ring density from DDS likely enhanced the energy transfer network for singlet and triplet states. Consequently, PSD capability initially increased then plateaued with increasing DDS content. The exact photophysical mechanism of DDS effects on PSD remains elusive.

Stability was assessed by remeasuring samples at 96 and 175 days post-synthesis. Figures 5(a)-(b) show relative LO and PSD values for all three measurement periods. Both PSD and LO trends with DDS concentration remained consistent across measurements. After six months, FOM values decreased by 8–19% and LO by 51% compared to day-2 measurements, indicating that stability requires further improvement. Figure 5(c) shows FOM dependence on Q (in MeVee) for the 30 wt% DDS scintillator (RK3), demonstrating effective neutron/gamma separation down to 150 keVee, where FOM = 1.27 meets the criterion for efficient PSD.

IV. SUMMARY

Organic scintillators based on PS and PVT are widely used in applications from nuclear physics experiments to safeguard equipment. However, their poor radiation resistance remains a major drawback. Cleavage of C–C and C–H bonds generates free radicals and hydrogen release, causing yellowing and reducing light yield and transparency. One solution involves replacing or partially substituting these materials with more radiation-hard polysiloxane polymers. In this work, we successfully developed a new elastic scintillator by incorporating 20–40 wt% of the siloxane compound DDS into a PVT matrix for neutron/gamma discrimination. The resulting organosilicon-loaded plastic scintillators comprise a PVT/siloxane-based matrix that is transparent, mechanically robust enough for machining and polishing, and provides excellent neutron/gamma discrimination. Characterization showed that DDS addition imparts elasticity and improves dye solubility by preventing aggregation. While achieving comparable PSD performance, the scintillators exhibit 76–101% LO relative to three-year-old EJ-200. However, increasing DDS concentration reduces light yield, suggesting that radioluminescence in DDS-PVT mixtures is less favorable than in pure PVT scintillators. Measurements at different times post-preparation indicate that PSD and LO stability need further improvement. The precise spatial conformation governing singlet and triplet exciton migration for efficient PSD in DDS-doped scintillators remains unknown. Future work will investigate cross-linked networks from copolymerization and homopolymerization processes. Nevertheless, this newly designed organosilicon-loaded plastic scintillator shows promise for

fabricating next-generation neutron detectors for high-dose irradiation environments.

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All authors contributed to this work. Weirong Liu and Pengcheng Shi designed and prepared the scintillators. Weirong Liu, Zhiyun Zhu, and Siyu Zhang performed the experimental measurements. Weirong Liu and Pengcheng Shi analyzed the data and wrote the manuscript draft, with all authors providing comments on subsequent versions. All authors have read and approved the final manuscript.

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