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Studying of Thermal and Shielding Properties of PVDF (PolyVinyliDene Fluoride) Pipes used in Aqueous Solution Sector of the Nuclear Fuel Fabrication Facility

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ABSTRACT

As a result of a fire or explosion in nuclear fuel fabrication facilities two risks to health and safety. Mitigation of fire hazards and radioactive release necessitates the use of fire-resistant and appropriate shielding materials, specifically plastic tubes used in chemical reactions during the conversion process. This paper aims to study the thermal stability, flammability, and shielding properties of polyvinylidene fluoride (PVDF) tubes used in the aqueous solutions sector of the Egypt Fuel Manufacturing Pilot Plant (FMPP), to can replacement the consumed one by another from the local market with the same property. For this purpose, PVDF samples were tested by Energy Dispersive X-ray spectroscopy (EDX) and X-ray direction (XRD). Thermal analysis tests as thermo-gravimetric analysis (TGA) as well as the derivative of thermogravimetric analysis (DTGA). The ignition test was applied using a limiting oxygen index (LOI), flame chamber (UL/94). ^{137}Cs radioactive source of 5mCi activity was used for gamma shielding properties measurement. Shielding parameters of fast neutrons like removal cross-section and mean free path was calculated. Results of (TGA), showed that PVDF has good thermal stability, where weight loss (about1%) is observed at a temperature above 422°C. PVDF sample successfully passed the UL-94 H-0 rating with a Limiting Oxygen Index (LOI) of about % 44.8. Also, it has appropriate shielding properties due to the high content of fluorine atoms. PVDF containing Al metal in their composition is more effective for the attenuation of fast neutrons.

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Introduction

At nuclear fuel fabrication facilities, criticality, explosion, fire, and release of radioactive materials are the primary risks. Hence the public and the environment should be protected using adequate design and construction and by safe operation [1]. Leaks from equipment such as pumps, valves, and pipes may lead to the dispersion of radioactive material and toxic chemicals, and the unnecessary generation of waste. Leaks of flammable gases or liquids can lead to explosions or fires. Chemical, toxic, flammable, or explosive substances can affect facility safety. The chemical toxicity of uranium in a soluble form such as UF_6 is more significant than its radiotoxicity [2]. Fire in uranium fuel fabrication facilities may lead to the dispersion of radioactive material or toxic material by breaching the containment barriers or may cause a criticality accident by affecting the system or the parameters used for the control of criticalities such as the moderation control system or the processing equipment dimensions. The operating organization shall make arrangements for ensuring fire safety based on a fire safety analysis. Such arrangements shall include, control of combustibles and ignition sources by the licensing documentation. The choice of tubes and valve material used for a chemical process must take into account the operating requirements. The selected material should have thermal stability, mechanical, fire resistance, and good radiation attenuation or absorption. Chemical and thermal considerations become more important [3]. The primary hazards from the release of UF_6 are chemical, while the radioactivity aspects present secondary hazards (chronic effects) [4]. Shielding is an important factor to protect humans and the environment from the effects of radiation. Polymers can easily be doped with high atomic number (high-Z) materials additives to form their composites that are more competent radiation shields [5-6]. In the nuclear industry, UF_6 is handled in the three physical states during the processing stages of the fuel cycle [7]. Teflon and fluoranthene are suitable for processing UF_6 in a hot aqueous solution at about 200°F due to their corrosion resistance properties [3]. Egyptian Fuel Manufacturing Pilot Plant (FMPP), which manufactures the nuclear fuel needed to operate the second Egypt Research Reactor (ETRR-2). According to the liquids handling, the circuit is divided into three sectors, UF_6 lines, aqueous solutions lines, and auxiliary lines. For the aqueous solutions sector all equipment and lines which handle HF, NH_4F, and ADU aqueous solutions are made of Teflon and PVDF, and valves are made of PVDF. It has excellent resistance to most chemicals including solvents and acids. High crystalline unreinforced fluoropolymer combines good mechanical, thermal, and electrical properties. Whereas fluoropolymers are rigid and resistant to deformation under mechanical stress and load [8], it also shows good resistance to high-energy radiation (considerably better than other fluoropolymers). Furthermore, its high elasticity, relative transparency, and ease of processing make this material suitable for various technological applications [9,10]. The most common applications of PVDF include nuclear waste processing, pipe, and nuclear industries [11-12]. The poly (vinylidene fluoride) - PVDF is a linear, semicrystalline homopolymer formed by the repetition of –(CF2–CH2) n – monomers, commonly used as a matrix due to its pyro and piezoelectric properties, as well as its

excellent chemical stability and mechanical resistance, combined with easy processing of this material. PVDF has good flame-retardant properties, when it burns it quickly self-extinguishes once the heat source is removed, preventing the spread of any remaining flames [13-15]. One of the most radiation-resistant polymers in the market is the poly (vinylidene fluoride) (PVDF) homopolymer. To overcome the risk of fire accident or to mitigate the consequences of fire, Prevention or stopping combustion could be by using flame retardant products [16]. The main parameter for polymer ignitability is the Limiting Oxygen Index (LOI). This determines the minimum oxygen concentration in the air for polymer ignition and burning. Air normally contains 21% oxygen, so polymers with LOI > 2I% will not burn in normal air. In practice, materials with at least 25% LOI should have lower ignition and burning [17]. For fuel cycle facilities, the expected properties of materials at the end of their useful life must be studied, due to the range and characteristics of the chemical and radiological conditions in normal operation and accident conditions. This may require provisions in the design for the monitoring of materials whose mechanical or thermal properties may change due to stress, erosion, chemical corrosion, or the induction of changes by irradiation [7]. In this work, studying the thermal, flammability properties and radiation attenuation capability of PVDF which is used as a pipe in the aqueous solution of the FMPP was carried out with direct gamma-ray exposure shielding features and shielding parameters of fast neutrons like removal cross-section and mean free path was calculated. This work aims to identify the possibility of replacing the aged pipes with another from the local market with same properties fulfil the safety requirements.

Material and Method

Polyvinylidene fluoride (PVDF) which is used as a pipe in the FMPP is supplied by the Argentine company Investigacion Aplicada (INVAP) was the material investigated in this study and compared with pure PVDF at Egypt marketing. The sample was used without further preparation or any change.

Energy Dispersive X-Ray Spectroscopy (EDX)

Scattering SEM/EDX, model EVO 60, with 10KV accelerating voltage, (central lab – Egyptian Nuclear and Radiological Regulatory Authority) was used for the elemental analysis or chemical characterization of a PVDF investigated sample

X-Ray Diffractions (XRD)

Shimadzu XRD 6000 diffractometer with Cu target was used. The XRD runs were carried out from10° to 40° at a scan speed of 8°/min. The XRD pattern for the investigated sample PVDF composite was measured using XD-DI Series, Shimadzu apparatus with a copper target (k ¼ 1.542 Å) at 40 kV operating voltage and an electric current of 30 mA.

Thermo-Gravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) evaluates the thermal stability of the composite samples.
(TGA) for PVDF samples was performed using the Shimadzu system. The tests were performed in a nitrogen atmosphere of 20 mL/min in the temperature ranges from ambient to 600 °C at a heating rate of 20 °C/min. Both peak maxima and temperature for weight loss were measured to characterize the thermal stability of the PVDF sample.

Fire Test

Flame chamber (UL94H) and LOI tests for PVDF samples were measured at the Fire and explosion protection laboratory, Egyptian National Institute of Standards.

Ignition Behavior (UL/94)

Horizontal burning rate measurements were performed using the UL94-H chamber according to ASTM D635-18, the test method for the rate of burning for PVDF samples in a horizontal position. The sample was with dimensions of 13 cm, 2.5cm, and 3 mm (length, width, and thickness) respectively. The test occurred with humidity of 45% and Room temperature of 21.1°C environmental conditions. The sample was exposed to a flame of 20 ±2 mm height horizontal position with ang 45° 30 ±1s exposure time. The time consumed and burning distance of the sample were recorded. The average burning rate was calculated based on the standard.

Limiting Oxygen Index (LOI)

LOI burning test was carried out at room temperature, and the samples were held vertically in an oxygen index system instrument, which was purchased from Fire Testing Technology Company, UK. The test was carried out at room temperature and humidity of 55.4% using an oxygen index instrument. The measurement was executed according to the standard test method ISO 4859-2. Propane gas was used as the flame source. The sample was subjected to flame in an atmosphere of (O_2, N_2) mixtures to determine the initial concertation of oxygen. The lowest visible part of the flame was applied to the top of the specimen using a sweeping motion to cover the whole surface. The samples were exposed to the flame for 30 ±1s. The oxygen concentration in (O_2, N_2) mixtures that cause the sample to be ignited for 5 cm or a period of the burning of 3 min as soon as the removal of the flame was considered the limiting oxygen index (LOI).

Shielding Measurements

Gamma-Ray Attenuation Measurement

^{137}Cs radioactive source of 5mCi strength was used. The incident and transmitted gamma-ray intensities were measured for a fixed preset time by recording the corresponding counts, using the3"x3" NaI (Tl) detector at the Egyptian Nuclear and Radiological Research Safety Center Atomic Energy Authority. The transmission experiment setup is shown in Figure 1.

The linear attenuation coefficient m [cm^{-1}] of the absorber has been calculated according to equation (1) [18].

$$\mu = \frac{1}{X} ln\left(\frac{I_0}{I}\right) \quad (1)$$

where I_0 is the detector count rate without the polymer sample, I the count rate with the investigated sample, and x [cm] is the sample thickness.
The mass attenuation coefficients, (μm)μ/ ρ = (cm^2 g^{-1}), were calculated according to Eq. (2), which is based on the Lambert-Beer law [19]

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The mass attenuation coefficients, (μ_m)μ/ ρ = (cm^2 g^{-1}), were calculated according to Eq. (2), which is based on the Lambert-Beer law [19]

$$\mu_m = \frac{\mu}{\rho} = \frac{1}{\rho X} \ln\left(\frac{I_0}{I}\right) \quad (2)$$

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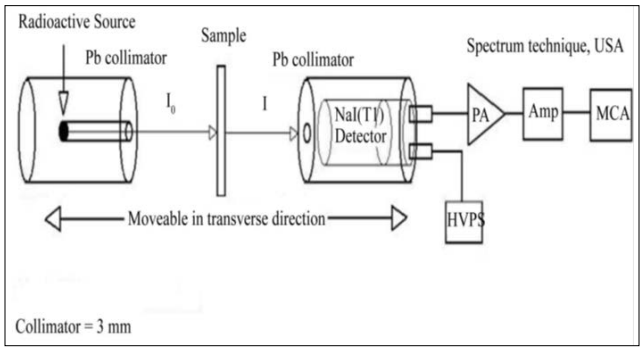

Figure 1: Gamma Transmission Experiment Set Up

The measured result is compared with the theoretical calculation using XCOM software which carries out to generate the cross-sections and attenuation coefficients for any element, compound, or mixture, at energies between 1 keV and 100 GeV. The thickness of an absorber reduces the radiation level to half of its initial level (HVL), is determined by the following equation:

$$HVL = (ln2/\mu) = (0.693/\mu) \quad (3)$$

Correspondingly the thickness required to reduce the radiation level to one-tenth of its initial value is defined as (TVL), was calculated using the following equation

$$TVL = (ln10/\mu) = (2.3026/\mu) \quad (4)$$

HVL and TVL are two important parameters that must be considered in studying new shielding material.

Radiation Protection Efficiency (RPE), is an important parameter to knowing a material's shielding ability and is determined as shown in Equation 5 [20]

$$RPE = (1-\frac{I}{I_0})\ 100 \quad (5)$$

Fast neutron effective removal cross-section (ΣR)

The method for calculating the attenuation of the fast neutron can be achieved by using the macroscopic effective removal cross-section (ΣR). (ΣR) represents the probability of the reaction of the neutron which is subject to the collision [21]. The effective removal cross-section for compounds or mixtures may be calculated using the value ΣR(cm^{-1}) or mass removal cross-section ΣR/ρ (cm^2g^{-1}) as in Eqs. (6) and (7) respectively [22-23]

$$\Sigma_R = \Sigma_i \rho_i (\Sigma_R/\rho)_i \quad (6)$$

$$\Sigma_R/\rho = 0.206\ A^{-1/3} Z^{-0.294} \quad (7)$$

Where ρ_i is partial density, which is calculated using Equation 8.

$\rho_i = F_i \rho_s$ (8)

Where ρ_i is the partial density of the compound as it appears in the sample, F_i: Fraction by Weight, and ρ_s the density of the sample. The reciprocal of the macroscopic cross-section is the mean free path λ.

$\lambda = 1/\sum_t$ (9)

For materials with a high absorption cross-section, the mean free path is very short, and neutron absorption occurs mostly on the material's surface, it is called self-shielding because the outer layers of atoms shield the inner layers.

Result and Discussion

Energy Dispersive X-ray Spectroscopy (EDX)

PVDF component and percentage using EDX techniques are represented in Figure 2. It is obvious that PVDF sample contains 0.37 wt % Al metal.

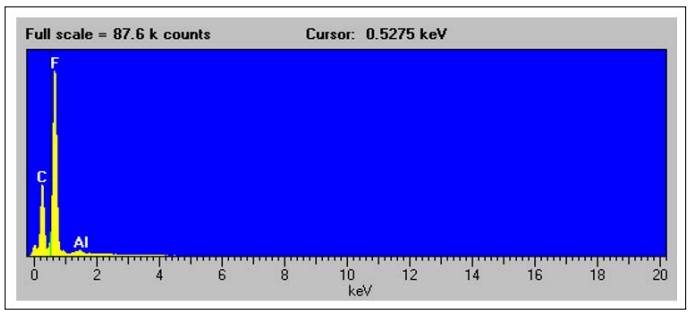

Figure 2: PVDF Components

X-Ray Diffraction

Figure 3 shows the XRD pattern of the PVDF investigated sample. PVDF has four known phases, named α, β, γ, and δ. Compared to pure PVDF, the peaks at 2θ =18.2, 20.04, and 26.5° correspond to the α-phase [24]. The peak at 26.5° corresponds to the α-crystalline phase and is shifted towards a higher region (28.30°) which is assigned to the β-crystalline phase, Aluminum additives increase the β-crystalline phase of PVDF. The peaks for PVDF samples with Al metal are sharper than pure PVDF samples in the literature [25]. The peaks at 38. 4° and 45.7° correspond to the Al element.

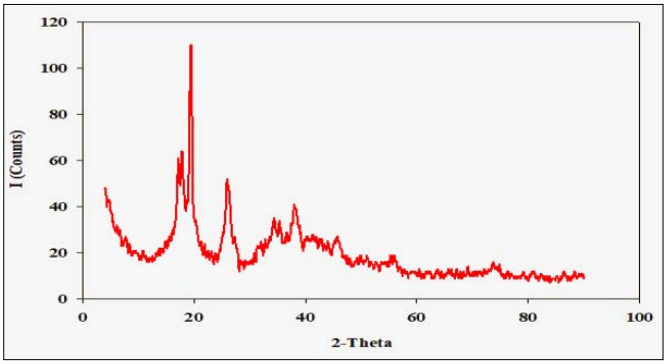

Figure 3: The XRD Pattern of the PVDF Sample

Thermal Stability Analysis

Figures 4 a and b represent (TGA) and (DTG) curves of PVDF respectively. The melting point is about 170 °C with no mass change in the TGA curve. A small weight loss (about10%) is observed at a temperature above 422ºC. The onset degradation temperature of the polymer chain for pure PVDF is around 400 ºC [26, 27], wherein the studied sample is around 420 ºC, that's due to the existence of the Al element [28]. When the temperature rises to 450–575°C, weight loss of 20% occurs at a lower peak with a temperature of about 468 after 44 minutes from the beginning of the experiment and the heights peak with weight loss of about 75% occurs at temperature 518°C after 55 minutes. From Figure 5b, DTGA curves, PVDF has a single degradation step with a high maximum decomposition temperature (T_{max}) of about 515 °C. The main exothermic peak appears at 555 ºC ahead of the melting point of Al (about 660 ºC), due to the decomposition of PVDF. The char residue reached 15% at 600 °C, the maximum decomposition rate temperature was about 574.5 ºC. All the reactions happen below the melting point of Al, in agreement with the literature [29]. The analysis shows that the existence of small amount of Al metal enhancement the thermal stability of PVDF with 20% greater than pure PVDF, which is around 400 ºC [27]. Consequently, the thermal stability and char residue of PVDF at high temperature was enhanced.

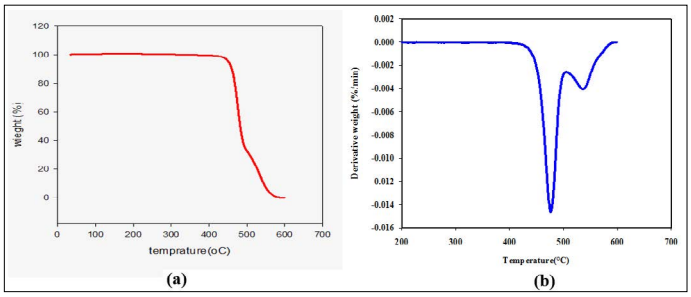

Figure 4: PVDF sample a. (TGA) and b. (DTA) curves

Flammability Behavior

UL94H test) horizontal burning rate test (presented that, the PVDF sample achieved H0 (mm/min) class without dripping. The image captured from the burning test is shown in Figure 5. The LOI value reached 44.8%, and LOI for pure PVDF with the same thickness was 43% [24]. This means that the existence of a small percentage of Al metal enhanced the flammability properties of PVDF. Aluminum catalyzes the dehydrofluorination of polyvinylidene fluoride at a temperature higher than 200 °C [30]. The dehydrofluorination process significantly increases the fraction of planar chain conformation in the PVDF with a wider processing temperature range, than pure PVDF. Then it does not support combustion under normal atmospheric conditions. It was consistent with the TGA results discussed above. According to LOI and UL94H data, the PVDF sample presented the best flame retardancy.

Figure 5: PVDF Sample after Burning Test

PVDF Sample Shielding Properties

The measured values of the linear attenuation coefficient μ (cm^{-1}) and mass attenuation coefficient μm (cm^2/g), were compared with calculation values obtained using the XCOM software, for PVDF samples. Table 1 illustrates the measured and calculated results of the linear and mass attenuation coefficients for photon energy 4.5 Mev. It is clear that the calculated and X-com values of μ and μm are in good agreement. The attenuation coefficients and mass attenuation coefficient μm (cm^2/gm) for PVDF with Al metal additives are greater than that in the case of pure PVDF as shown in Table 1. Both HVL and TVL values for PVDF/Al decrease more than their values for the pure PVDF sample, due to the increase in material density. It is indicated that the density of the sample is an important parameter in the attenuation calculation. The gamma-ray shielding efficiency for the PVDF/Al sample with 3mm thickness was about 36.3%. The mass attenuation coefficient for PVDF/Al and pure PVDF versus photon energies from 0. 1 MeV to 100 MeV was calculated using the X-com program. The results had been illustrated in Table 2 and Figure 6 respectively. From Figure 6 it is clear that in the low energy region, Eg < 0.5 MeV where μ_m decreases sharply, the photoelectric effect is dominant, and in the high energy region, Eg> 0.5 MeV, where μ_m decreases slightly, whereas the Compton scattering overlaps with the photoelectric effect. By increasing the gamma-ray energy the interaction cross-sections are decreased and pair production is significantly higher [31]. It is obvious that the calculated μ/ρ for PVDF/Al sample is higher than pure PVDF. This is attributed to photoelectric absorption on the elemental composition, the higher effect of atomic number, and composites density. Then the addition of a small amount of aluminum metal enhanced the gamma radiation shielding by about 15%. These values are useful for selecting the best radiation shielding PVDF that has a higher content of Al metal.

Table:1 Measured and calculated values of linear attenuation and mass attenuation coefficients for photon energy 4.5 Mev

sample	Experimental calculation				Theoretical using Xcom			
	(cm-1)	(cm^2/gm)	(cm)		(Cm^{-1})	(cm^2/gm)	(cm)	
	μ	μ_m	HVL	TVL	μ	μ_m	HVL	TVL
Pure PVDF	0.054	0.031	12.84	42.64	0.0502	0.029	13.81	45.9
PVDF+ Al metal	0.073	0.0366	9.5	31.54	0.070	0.0354	9.90	32.9

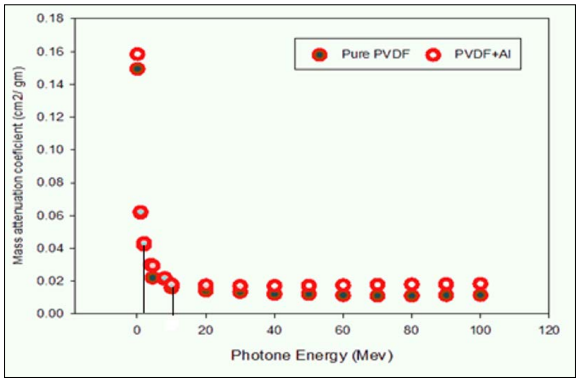

Figure 6: Total Mass Attenuation Coefficients for PVDF Samples with Different Photon Energy

Table 2: The Theoretical Values Mass Attenuation Coefficients, Estimated by the Xcom Program of PVDF and PVDF/Al

Photon energy (MeV)	Total mass attenuation coefficients for samples (μ/ρ) cm^2/gm	
	PVDF	PVDF/Al
1.00E-01	1.55E-001	1.59E-001
1.00E+000	6.37E-002	6.30E-002
2.00E+000	4.45E-002	4.41E-002
4.00E+000	3.38E-002	3.59E-002
4.50E+000	2.9E-002	3.48E-002
8.00E+000	2.23E-002	2.29E-002
1.6 E+001	1.78E-002	1.90E-002
2.0 E+001	1.71E-002	1.84E-002
3.0 E+001	1.63E-002	1.80E-002
4.0 E+001	1.61E-002	1.80E-002
5.0 E+001	1.62E-002	1.82E-002
6.0 E+001	1.63E-002	1.84E-002
7.0 E+001	1.64E-002	1.87E-002
8.0 E+001	1.66E-002	1.89E-002
9.0E+001	1.67E-002	1.91E-002
1.00E+002	1.69E-002	1.93E-002

Table 3 represents the calculated values for $\Sigma R/\rho$, ΣR, and λ. It is clear from the table that with Al existence $\Sigma R/\rho$ increase, where the weighted fraction of the added element will increase the contribution of each element in determining the value of the total cross sections will be increased for all compounds. Otherwise mean free path decreases due to an increase in the density of the sample by adding Al, reinforcing the sample where the distance traveled by the neutron inside the shield decreases.

Table 3: Calculated Values of Mass Removal, Removal Cross-Sections and Mean Free Path for Pvdf Samples

Compound	$\Sigma R/\rho$ (cm^2g^{-1})	Density (gm/cm^3)	ΣR (cm^1)	λ (cm)
PVDF $C_2H_2F_2$	0.194	1.79	0.3453	2.9
PVDF/ Al	0.2263	2.04	0.4617	2.17

Conclusion and Recommendation

Polyvinylidene fluoride (PVDF) is a semi-crystalline polymer having thermal stability. In the field of nuclear fabrication facilities, a high flame-retardant grade is required. PVDF/Al successfully passed the UL94H test with a rating of H0 and an LOI test value of 43.8%. Studies concluded that PVDF tubes have good thermal stability, as thermogravimetric analysis (TGA) the sample is thermally stable up to 425°C while (TGA) for pure PVDF is thermally stable up to 375°C. However, PVDF has good thermal stability and exhibits good durability when exposed to continuous irradiation. It was demonstrated that the existence of Aluminum metal with a small percentage of the PVDF, reduced the combustion intensity, and improved the residue char at the end of burning. Also improves its radiation-shielding properties. The shielding parameters of the PVDF with Al compared to pure PVDF shielding materials. The comparison showed that the shielding parameters PVDF/Al metal are about 15% more than pure PVDF. Shielding efficiency for the PVDF sample with 3mm thickness was about 36.3%. The results show that fast neutron removal cross-section depends on the density and chemical composition of the shielding materials. The sample containing Al in its composition is more effective for the attenuation of fast neutrons than the pure sample. The study shows that PVDF can retain its physical properties with ignition delay during fire accidents. Aged PVDF tubes can be replaced with tubes from the local market, considering that are no less than these properties.

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Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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