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Temperature- and frequency-dependent ultrasound response of a Poly(N-vinylcaprolactam) polymer gel dosimeter

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Abstract

Poly(N-vinylcaprolactam) (PNVCl) polymer gels have recently been proposed for radiation dosimetry; however, their ultrasound response remains inadequately defined. This study assessed the feasibility of pulse-echo ultrasonography for evaluating the dose response of a PNVCl polymer gel dosimeter. Gel samples composed of gelatin type A, deionized water, BIS, PNVCl, and tetrakis(hydroxymethyl)phosphonium chloride (THPC) were synthesized and irradiated with 6 MV photon beams. Dose-response measurements were performed from 0 to 30 Gy at 200 cGy min^{-1}, and dose-rate dependence was assessed at 100, 300, and 600 cGy min^{-1} for doses of 5 and 10 Gy. Ultrasound measurements were acquired with transducers operating between 1 and 4 MHz. The speed of sound and attenuation coefficient were determined using pulse-echo time-of-flight and frequency-domain analyses, respectively. All ultrasonic parameters increased with absorbed dose, with the clearest linear response observed at 2 MHz. The response was temperature dependent, with higher sensitivity at room temperature than at 15 °C. Dose rate and photon beam energy showed no clear influence on the ultrasonic response within the investigated range. These findings support pulse-echo ultrasonography as a practical method for assessing PNVCl polymer gel dosimeters, provided that temperature is carefully controlled and temperature-specific calibration curves are used.

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Keywords: Poly(N-vinylcaprolactam) polymer gel, ultrasound dosimetry, speed of sound, attenuation coefficient

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1. Introduction

Nanogels are three-dimensional networks of nanometrically cross-linked polymers that combine the advantages of nanotechnology with the hydrophilicity and flexibility of hydrogels [1, 2]. They are potential drug carriers because of their large surface area, porous structure, biocompatibility, and readily functionalized surfaces [3]. Smart nanogels change their structural characteristics and hydrophilic–hydrophobic balance in response to internal or external stimuli [4, 5].

Ultrasound is a non-ionizing imaging technique that uses high-frequency sound waves to examine tissue structure and function by analyzing reflected echoes, transmitted signals, and Doppler frequency shifts [6–10]. It is widely used for anatomical imaging, blood-flow assessment, and guidance of interventional procedures because it is real time, portable, relatively low cost, and does not use ionizing radiation [11–16]. In radiotherapy, ultrasonography has become an important tool for image guidance and motion management, particularly in regions

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requiring soft-tissue visualization without additional radiation exposure. Ultrasound facilitates patient positioning and target localization, aids the monitoring of intra-fraction organ motion, and supports adaptive workflows by enabling repeated imaging during treatment [17].

Poly(N-vinylcaprolactam) (PNVCl) is a water-soluble polymer with a lower critical solution temperature (LCST) in aqueous environments [18]. Related macromolecular systems include N-alkylated poly(acrylamides) [19–21], methylcellulose [22, 23], poly(methyl vinyl ether) [3], and block copolymers of ethylene oxide [21, 24]. These polymers and their derived hydrogels have attracted increasing interest in biotechnology and biological applications [25–30]. In this context, PNVCl has properties that can support the encapsulation of enzymes and whole cells.

Among stimulus-sensitive drug carriers, heat-sensitive polymers such as PNVCl undergo a phase transition of their cross-linked structure in water when the temperature exceeds a critical threshold, known as the transient volume phase transition (TVPT). This thermo-responsiveness is important because it can be adjusted toward human body temperature through copolymerization with hydrophilic comonomers, such as poly(ethylene glycol) (PEG), enabling temperature-controlled drug release [31, 32]. In addition, PNVCl is biocompatible, and potential hydrolysis of the amide group of NVCL may yield a polymeric carboxylic acid that is suitable for biomedical purposes [4, 21, 33].

The amphiphilic, organo-soluble N-vinylcaprolactam monomer dissolves in non-polar and polar organic media. PNVCl synthesis commonly uses free-radical initiation at high temperatures in solvents such as benzene [34, 35], isobutanol [36], or isopropanol [37, 38]. Bulk and microemulsion polymerizations of PNVCl have also been reported [39, 40]. The latter synthesis occurred above the melting point of PNVCl, resulting in monomer melting [41]. PNVCl synthesis in aqueous media is required for the preparation of copolymers with insoluble or poorly soluble comonomers, chemically cross-linked hydrogels, and enzyme- or cell-immobilization systems [42].

The objective of this study was to assess the dose-response characteristics of a PNVCl polymer gel dosimeter using pulse-echo ultrasound. Variations in sound velocity and attenuation coefficient were evaluated over a dose range of 0–30 Gy, and the effects of frequency, measurement temperature, dose rate, and beam energy were also investigated. The PNVCl polymer gel dosimeter was prepared using the method reported by Rabaeh *et al.* [43].

2. Methods

2.1. Preparation

The method of Rabaeh *et al.* [43] was used to prepare the PNVCl polymer gel samples, with a slight change in the THPC concentration. The gel formulation contained 326 mL of deionized water, 30 g of gelatin type A, 12 g of BIS, 12 g of NVCL, and 1.3 g of THPC (Table 1). All chemicals were obtained from Sigma-Aldrich.

Table 1. PNVCl polymer gel chemical composition.

Chemical	Concentration
Water	326 mL
Gelatin type A	30 g
BIS	12 g
PNVCl	12 g
THPC	1.3 g

Gelatin was mixed with deionized water at room temperature for 5 min. The mixture was then gradually heated to 50 °C for 60 min using a magnetic hot-plate stirrer (IKA, Germany) to obtain a homogeneous solution. BIS was added and mixed until dissolved. After the solution cooled to 37 °C, THPC was added and mixed for 2 min. Before irradiation, the gel solution was transferred into 50 mL plastic containers and stored at 10 °C. This modification was based on observations by Rabaeh *et al.* [44], who reported a relationship between the time at room temperature and THPC concentration. To facilitate comparison with previous studies, the standard formulation of the NVCL polymer gel dosimeter was used [45, 46].

2.2. Ultrasound property measurements

The ultrasonic properties were measured using a PHYWE Ultrasonic Solids and Solutions Echoscope. After preparation, the gels were kept in a Petri dish under refrigerated conditions for 24 h to preserve structural integrity [47]. Samples were then measured after being equilibrated to the required measurement temperature. Transducers operating between 1 and 4 MHz were used. The transducers included snap-in couplings, and the device automatically identified their frequency. Variable transmission and reception powers were available [48].

Before ultrasound testing, samples were equilibrated to either room temperature or 15 °C. Equation (1) gives the expression used to determine the speed of sound by the pulse-echo (PE) technique [49]. In this method, the travel time of the ultrasonic wave from the transducer to the reflector and back is determined from the sent and received signals. The time of flight (TOF) was assessed by comparing corresponding peak signals. The TOF measurement may be influenced by the protective layer of the transducer; therefore, the measured value includes both the propagation time through the sample and the time required for the signal to pass through the protective layer [48, 50, 51]:

$$\mathrm{SOS} = \frac{2d}{t}, \tag{1}$$

where SOS is the speed of sound (m s^{-1}), d is the depth of the gel in the Petri dish (m), and t is the time of flight (s).

To evaluate attenuation, the frequency-dependent attenuation of each signal was calculated by applying a fast Fourier transform (FFT) to the radiofrequency (RF) signal from the reflector. After the speed of sound in the sample had been determined, the attenuation coefficient was calculated using the

A-scan pulse-echo method according to

$$\alpha = \frac{20 \log_{10}(A_1/A_2)}{2D}, \quad (2)$$

where α is the attenuation coefficient (dB cm^{-1}), A_1 and A_2 are the echo amplitudes before and after attenuation through the sample, respectively, and D is the sample thickness (cm). The factor of 2 accounts for the round-trip propagation path in pulse-echo measurements.

2.3. Irradiation

Before irradiation, gel samples were kept in the treatment room for 2 h to reduce temperature differences between the samples and the irradiation environment. The plastic containers were placed in a water phantom and irradiated at a depth of 5 cm, with a source-to-surface distance (SSD) of 100 cm and a field size of 10 × 10 cm^2. Irradiation was performed using a Siemens Primus linear accelerator (Germany).

In the dose-response experiment, samples were irradiated with 6 MV photon beams over a dose range of 0–30 Gy at a dose rate of 200 cGy min^{-1}. To evaluate dose-rate dependence, additional samples were irradiated to 5 and 10 Gy at dose rates of 100, 300, and 600 cGy min^{-1}. To evaluate beam-energy dependence, selected samples were treated using 15 MV photon beams. After irradiation, samples were stored at 10 °C for 24 h before ultrasound testing.

For each irradiation condition, three independent samples were measured and the results were averaged. Because full uncertainty propagation was not performed, this limitation is acknowledged. Future work should include repeated ultrasound acquisitions, detailed uncertainty estimation, and stricter temperature-controlled measurements.

2.4. Statistical analysis

Data are presented as the mean of three samples. Linear regression analysis was used to evaluate the relationship between absorbed dose and each ultrasound parameter at different frequencies and temperatures. The slope of the fitted line was used to describe sensitivity, and the coefficient of determination (R^2) was used to assess linearity.

3. Results and discussion

3.1. Ultrasonic speed of sound and attenuation coefficient at room temperature

At room temperature, both sound velocity and attenuation coefficient varied consistently with absorbed dose over the investigated range of 0–30 Gy. The clearest linear response for sound velocity was observed at 2 MHz. Ultrasound evaluation of polymer gel dosimeters has shown promise for assessing structural changes after irradiation, with acoustic speed, attenuation, and transmitted signal intensity exhibiting dose-related variation.

Figure 1 shows the relationship between sound velocity and dose at 2 MHz. The sound velocity in the irradiated gel increased over the investigated dose range. A strong relationship between sound velocity and absorbed dose was observed at room temperature, with the gel showing a sensitivity of approximately 101 m s^{-1} Gy^{-1} and R^2 = 0.85.

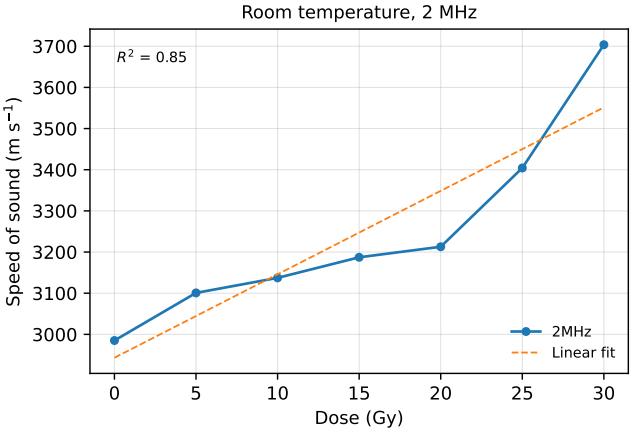

Figure 1. Dose response of ultrasound sound velocity in PNVCl polymer gel dosimeters measured at 2 MHz at room temperature over 0–30 Gy in 5 Gy increments.

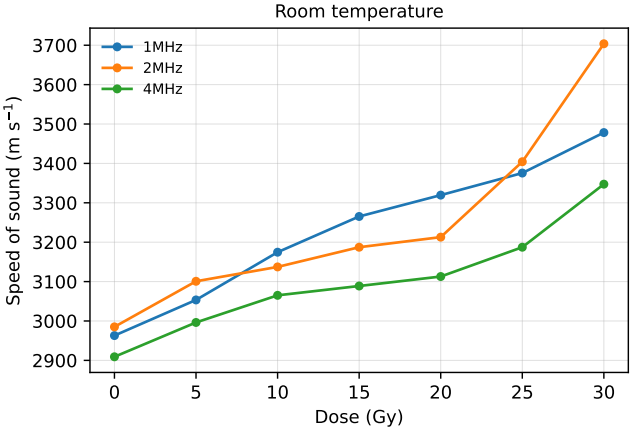

Figure 2. Effect of measurement frequency (1, 2, and 4 MHz) on ultrasound sound velocity in PNVCl polymer gel dosimeters at room temperature over 0–30 Gy.

The gel was measured at several frequencies to confirm the frequency at which sensitivity was greatest. Figure 2 shows that sound velocity increased with absorbed dose at all tested frequencies, although the response was clearest at 2 MHz.

The attenuation coefficient was measured using tone-burst pulses at 1, 2, and 4 MHz. The coefficient was determined from the slope of the graph relating transmitted amplitude to sample thickness for samples exposed to different doses. This approach reduces the influence of window attenuation. Figure 3 shows that the attenuation coefficient increased with dose for all transmitted frequencies from 5 to 30 Gy, indicating that irradiation increased gel sensitivity.

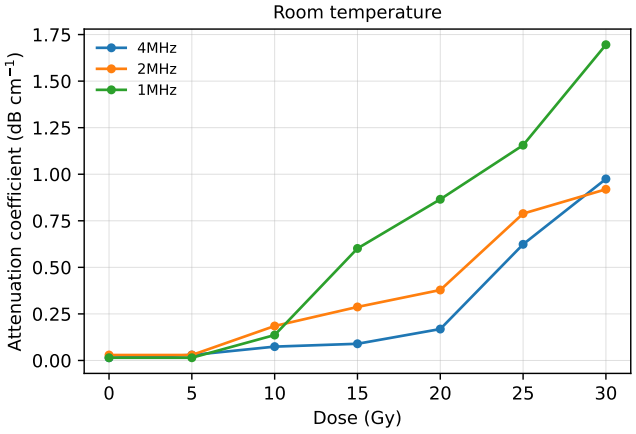

Figure 3. Dose response of the attenuation coefficient in PNVCl polymer gel dosimeters measured at 1, 2, and 4 MHz at room temperature over 0–30 Gy.

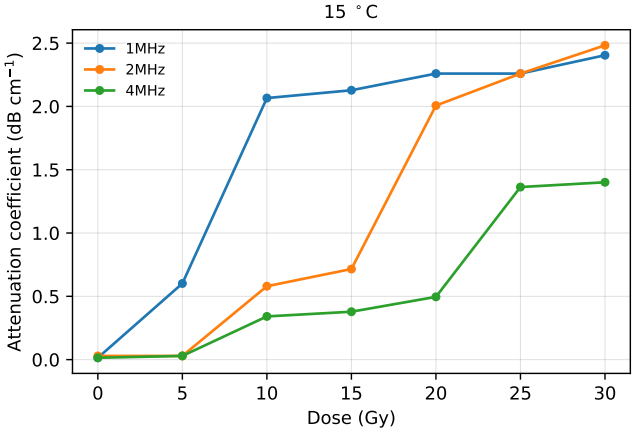

Figure 6. Dose response of the attenuation coefficient in PNVCl polymer gel dosimeters measured at 1, 2, and 4 MHz at 15 °C over 0–30 Gy.

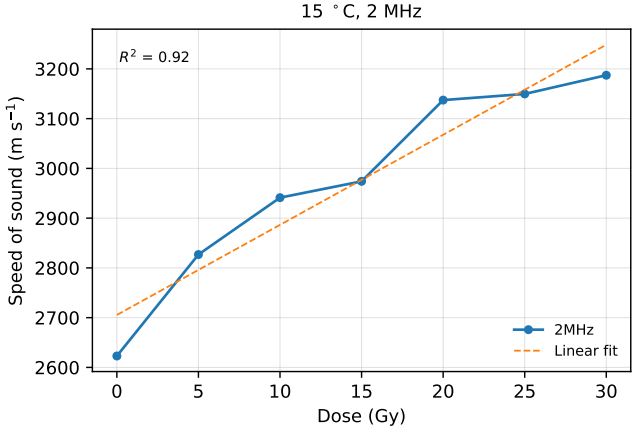

Figure 4. Dose response of ultrasound sound velocity in PNVCl polymer gel dosimeters measured at 2 MHz at 15 °C over 0–30 Gy in 5 Gy increments.

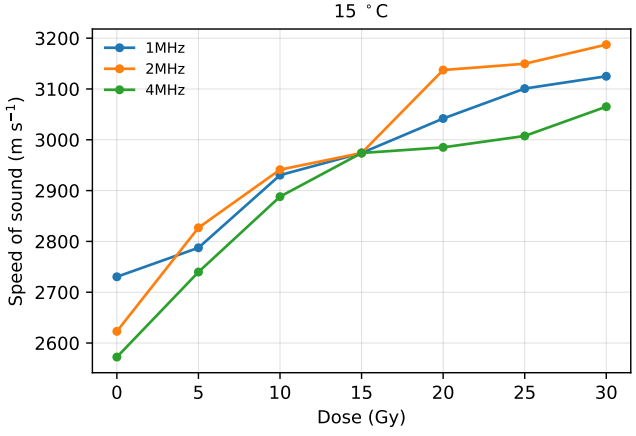

Figure 5. Effect of measurement frequency (1, 2, and 4 MHz) on ultrasound sound velocity in PNVCl polymer gel dosimeters at 15 °C over 0–30 Gy.

3.2. *Ultrasonic speed of sound and attenuation coefficient at 15 °C*

Figure 4 shows the relationship between sound velocity and dose at 15 °C and 2 MHz. The sound velocity increased approximately linearly with dose, with sensitivity of approximately 90.5 m s^{-1} Gy^{-1} and R^2 = 0.92. Compared with the room-temperature measurements in Figure 1, the sensitivity of the gel dosimeter was higher at room temperature than at 15 °C. Thus, the acoustic properties of this gel were more sensitive under room-temperature measurement conditions.

Measurements at different frequencies showed that the highest sensitivity was observed at 2 MHz. Figure 5 shows the increase in sound velocity at 1, 2, and 4 MHz. The increase in sound velocity with absorbed dose may indicate that density changes in the polymer gel dosimeter were more pronounced than changes in elastic modulus.

Figure 6 shows the relationship between attenuation and absorbed dose. Similar to the acoustic-velocity data, the attenuation response increased over the investigated dose range. The attenuation data indicate that the quasi-linear region extends to approximately 30 Gy, suggesting that attenuation may be useful for estimating absorbed dose.

Figures 1, 2, 4, and 5 show that increased dose sensitivity was associated with increases in both speed of sound and attenuation coefficient. However, the enhancement in ultrasonic properties at room temperature was greater than that observed at 15 °C. This may result from absorption caused by viscous limitations after transitions between adjacent sections in an object as temperature decreases [52]. Viscous losses may be linked to shear viscosity, which contributes to absorption even in purely longitudinal motion. Bulk or volume viscosity, which quantifies mechanical energy dissipated by a fluid under compression or dilation, may also contribute to sound absorption [52, 53]. In polymer gel dosimeters, scattering is considered negligible because the inhomogeneities are not resolved [54]. This is attributable to the ultrasound wavelength used, which may be several hundred times larger than the typical dimensions of inhomogeneities in the polymer gel dosimeter, potentially reaching

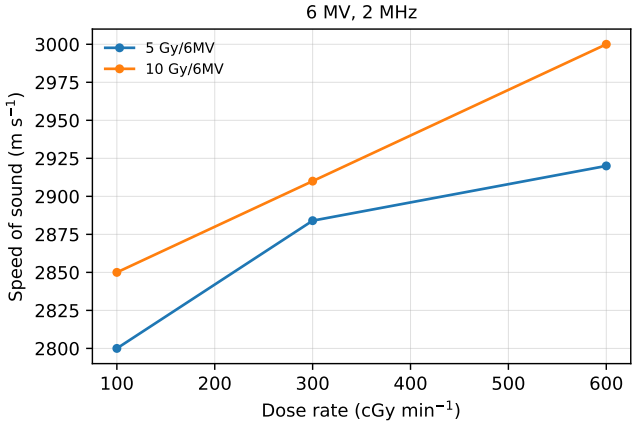

Figure 7. Effect of dose rate (100, 300, and 600 cGy min^{-1}) on ultrasound sound velocity in PNVCl polymer gel dosimeters irradiated to 5 and 10 Gy using 6 MV photons.

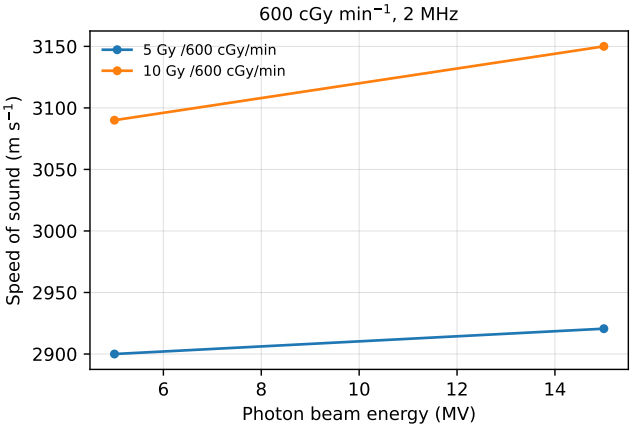

Figure 8. Effect of photon beam energy (6 and 15 MV) on ultrasound sound velocity in PNVCl polymer gel dosimeters irradiated to 5 and 10 Gy.

approximately 900 nm in diameter [55].

The measured speed-of-sound values may also be affected by irradiation-induced structural changes in the PNVCl gel matrix. As the polymer network becomes more cross-linked or more solid-like, the elastic stiffness of the medium may increase, leading to higher ultrasound propagation velocity. However, sound speed is not governed by stiffness alone; it also depends on density and the overall acoustic properties of the medium. Therefore, the observed behavior may reflect combined changes in polymer network structure, stiffness, and density after irradiation.

3.3. *Influence of dose rate on the PNVCl dosimeter*

Within the investigated range, changing the treatment dose rate from 100 to 600 cGy min^{-1} produced only small changes in sound velocity at either 5 or 10 Gy (Figure 7). No clear beam-energy dependence was detected between 6 and 15 MV (Figure 8). The data indicate that the ultrasonic response of the PNVCl gel was predominantly unaffected by dose rate and photon beam energy under the present experimental conditions.

Figure 7 indicates that small changes in dose rate had limited effect on the ultrasonic properties of the hydrogel dosimeter. These results suggest that the new hydrogel dosimeter can be irradiated at different dose rates without a major change in its response. This observation is consistent with the report by Rabaeh *et al.* [43].

Samples were also irradiated using 15 MV X-ray beams, as shown in Figure 8. The results indicate that changing the radiation beam energy at a dose rate of 600 cGy min^{-1} had no clear effect on the dose response of the irradiated PNVCl hydrogel. These findings suggest that the proposed PNVCl gel dosimeter may be a promising candidate for radiotherapy dosimetry.

Although the measured speed of sound showed relatively small variations across the investigated dose-rate range, the available data and associated uncertainties are insufficient to draw a definitive conclusion regarding dose-rate independence. Further studies with larger sample numbers, uncertainty analysis, and appropriate statistical testing are required to confirm whether the ultrasound response is dose-rate dependent or dose-rate independent.

3.4. *Theoretical energy dependence and water equivalency of the PNVCl polymer gel dosimeter*

To verify the radiological water equivalence of the PNVCl polymer gel dosimeter, density and photon mass energy-absorption coefficients were compared with those of water and soft tissue (ICRU 44) [56]. Theoretical attenuation coefficients were obtained from the NIST XCOM photon cross-section database, which provides photon interaction cross sections and attenuation coefficients for elements, compounds, and mixtures over the photon-energy range of 1 keV to 100 GeV [57].

The differences in photon mass energy-absorption coefficients among the PNVCl polymer gels, water, and soft tissue over 0.1–20 MeV were lower than 1.5%. This suggests that the proposed polymer gel dosimeter is nearly energy independent over 0.1–20 MeV and is approximately water and tissue equivalent. Accordingly, dose may be estimated using the proposed PNVCl system in radiation therapy without energy-dependent corrections.

3.5. *Comprehensive assessment and explanation of the results*

Ultrasound shows promise for evaluating radiation-induced structural changes in polymer gel dosimeters. In the 0–30 Gy range, acoustic velocity and attenuation changed with dose and demonstrated an approximately linear relationship with absorbed dose. Overall, ultrasonography provided a wide dose-response dynamic range while offering a relatively simple and potentially lower-cost approach for radiation dosimetry.

Mather *et al.* [55] used ultrasound to analyze radiation-induced changes in polymer gel dosimeters. Glass tubes were irradiated to 50 Gy using a Gammacell 200 Co-60 source, and the ultrasonic dose-response curves showed a much wider dynamic range than magnetic resonance imaging (MRI) data [55, 58]. Masoumi *et al.* [59] investigated the sensitivity of

the MAGIC-f polymer gel irradiated with 1.25 MV Co-60 in 2 Gy increments from 0 to 60 Gy. The reported sensitivities were 50 m s^{-1} Gy^{-1} for the dose–velocity curve and 0.06 dB MHz^{-1} Gy^{-1} for the dose–attenuation coefficient curve over the linear range of 4–44 Gy.

Radiation-induced polymerization and cross-linking within the gel matrix cause changes in ultrasonic parameters in polymer gel dosimeters. Ionizing radiation triggers chemical reactions and structural changes in polymer molecules, including cross-linking, thereby forming a more interconnected network. This makes the gel matrix more rigid and stable and affects mechanical properties and ultrasonic parameters such as attenuation and sound velocity. Radiation can polymerize gel molecules and change their density and acoustic properties. Radiation-induced changes may also affect gel swelling, porosity, density, and ultrasonic wave propagation [60, 61].

Energy transfer from radiation to a medium can change properties related to radiation absorption, thereby affecting ultrasonic parameters. As radiation dose increases, absorbed energy increases, leading to higher sound speed and attenuation coefficient because of radiation–material interactions. Radiation-induced structural changes or chemical reactions can increase a material's attenuation coefficient at higher doses. With increasing radiation exposure, the material's ability to attenuate radiation increases proportionally, indicating relatively homogeneous radiation absorption.

3.6. Uncertainty in the measurement of ultrasonic characteristics

Dose strongly affects the measured acoustic characteristics, which change as dose increases. Despite these promising results, variability must be reduced before ultrasonography can be used for absorbed-dose evaluation in polymer gel dosimetry. Variability may arise from acoustic-parameter measurement discrepancies and experimental conditions during sample assessment. Acoustic characteristics are affected by signal noise, information loss during digitization, and sampling errors during processing. In the present method, signals were repeatedly acquired and averaged to reduce noise, and the highest available sampling rate was used to reduce digitization error.

Temperature variations during experiments may affect acoustic characteristics. Studies performed in a temperature-controlled environment have shown that ±1 °C temperature variations can introduce uncertainty into acoustic properties [58]. This effect can be reduced by careful control of water and sample temperatures during experiments.

Another uncertainty factor is cuvette position. Although cuvettes are fixed at their base during measurements, tilting may alter the acoustic path length through the polymer gel and the angle of incidence at the cuvette surface. Both effects influence attenuation, while the path length through the polymer gel affects propagation speed. Improved cuvette-holder designs require further study.

Optimization of the ultrasound technique for polymer gel dosimeter evaluation requires minimization of ultrasonic measurement uncertainty. More sensitive transducers and optimization of polymer gel formulation may improve performance. Waterproof transducers that can be immersed in water tanks without acoustic coupling gel may also be beneficial.

4. Conclusion

The PNVCl polymer gel dosimeter demonstrated dose-dependent variations in ultrasonic sound velocity and attenuation coefficient over the range of 0–30 Gy. The optimum response occurred at 2 MHz, and measurements performed at room temperature showed greater sensitivity than those performed at 15 °C. Under the investigated experimental conditions, dose rate and photon beam energy did not significantly influence the ultrasonic response. These findings support the practicality of pulse-echo ultrasound for assessing PNVCl polymer gel dosimeters, provided that temperature is strictly controlled and temperature-specific calibration curves are used.

Data availability

All data generated or analysed during this study are included in this published article.

Declaration of competing 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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