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A Comprehensive Study on Bakelite Valves in Diaphragm Gas Meters and Improvement of their Physical and Mechanical Properties by Electron Beam Irradiation

Mustafa Mohammadi[1], Gholam-Reza Nejabat[1,2]*, M. Sadegh Tavallali[2,3] and Mehdi Rasti[4]

1. Department of Polymer Engineering, Shi.C., Islamic Azad University, Shiraz, Iran.

2. Department of Applied Researches, Chemical, Petroleum & Polymer Engineering Research Center, Shi.C., Islamic Azad University, Shiraz, Iran.

3. Department of Chemical Engineering, Shi.C., Islamic Azad University, Shiraz, Iran.

4. Fars Province Gas Company (FPGC), Shiraz, Fars, Iran

Abstract

The present study investigates the effects of electron beam irradiation on the physical and mechanical properties of Bakelite valves used in diaphragm gas meters. The irradiation is performed under vacuum at ambient temperature. Various analytical techniques, including FTIR, SEM, EDX, XRD, AFM, and TGA, are employed to monitor the physical characteristics of the samples before and after the treatment. In addition, mechanical properties, specifically impact and tensile behaviors, are assessed using an Izod impact tester and a universal testing machine (UTM). A 12 MeV electron beam is utilized at two doses: 60 kGy and 80 kGy. The results demonstrate that irradiation at 60 kGy results in significant enhancements in both physical and mechanical properties. In contrast, at 80 kGy, while some properties exhibit slight improvements, others show deterioration. Moreover, FTIR analysis reveals the elimination of hydroxyl groups at both irradiation doses. In addition, SEM and AFM analyses confirm that the surface properties of samples irradiated at 60 kGy are improved. Furthermore, XRD shows a decrease in crystallinity at this dose. Also, TGA results indicate that samples irradiated at 60 kGy possess higher thermal stability. Specifically, samples irradiated at 60 kGy show a 180% increase in impact strength, whereas those at 80 kGy show a 55% increase. Moreover, the Tensile strength increases by 2% for samples treated at 60 kGy, whereas it decreases by 34% for those treated at 80 kGy. Ultimately, the results confirm that upon electron beam irradiation of Bakelite valves, crosslinking dominates over degradation at 60 kGy, while degradation becomes dominant at 80 kGy.

Keywords: Bakelite Valves, Diaphragm Gas Meters, Electron Beam Irradiation, Phenol Formaldehyde.

Introduction

Phenol formaldehyde resins (PF), also called phenolic resins, are a highly desirable group of thermosetting polymers, recognized for their excellent physical and mechanical properties. They offer several advantages over conventional thermoplastics and other thermosetting resins, including exceptional adhesive qualities, high rigidity, excellent dimensional stability at elevated temperatures, superior thermal properties, remarkable mechanical strength, durability, outstanding flame resistance and retardancy, effective heat insulation, a highly cross-linked structure, efficient glue-bond formation, and strong chemical stability [1].

Bakelite, a specific type of PF resins, retains its shape and dimensions even when subjected to heating, cooling, or exposure to various chemicals and solvents. These attributes, combined with its lightweight nature and low electrical conductivity, have led to extensive applications in multiple fields, including the automotive and electronics industries, household products, construction, various industrial tools, and particularly in the internal valves of gas meters [1-3].

The chemical structure of Bakelite powder resembles that of Novolak resin, which is formed through the polycondensation of phenol and formaldehyde in phenol-rich reactions under highly acidic conditions (pH=1-4). This resin is known for being soluble and fusible with molecular weights in the range of 200 to 2000 Da [4].

**Corresponding author:* Gholam-Reza Nejabat, Department of Polymer Engineering, Shi.C., Islamic Azad University, Shiraz, Iran

Department of Applied Researches, Chemical, Petroleum & Polymer Engineering Research Center, Shi.C., Islamic Azad University, Shiraz, Iran

E-mail addresses: gr.nejabat@iau.ac.ir, ghnejabat@yahoo.com

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Moreover, when a base catalyst such as hexamethylenetetramine is used, stepwise condensation reactions can occur between the resin molecules. In the first two steps of this process, condensation and the removal of water take place. By the end of the second step, an insoluble but swellable Bakelite powder is produced, which can be compounded and molded under pressure [4]. Also, Bakelite can be produced from resoles, formed in formaldehyde-rich reactions under basic conditions (pH > 7). These resoles are then cured by applying heat and pressure to yield Bakelite [4]. During the compounding and molding stages of the phenolic (PF) powders, various additives, including graphite powder, carbon black, wood powder, calcium carbonate, and asbestos, may be included. The curing process is then carried out at relatively high temperatures and pressures, resulting in an infusible and insoluble molded Bakelite piece [4, 6]. Additionally, the finished products may undergo post-curing processes to minimize shrinkage and ensure dimensional stability for future applications [7].

The Bakelite internal valves and valve seats in diaphragm gas meters play a crucial role in accurately measuring gas volume. Any defects in these components can lead to miscalculations in both received and delivered gas volumes, resulting in internal gas leaks and economic losses due to unaccounted gas phenomena [8]. Moreover, industry experts have identified several factors that contribute to the malfunction of these Bakelite valves, including abrasion, cracks, crevices, torsion, and swelling. In addition, these issues may be linked to the materials used, particularly the types and amounts of fillers, as well as the production process, such as the post-curing step. Addressing these weaknesses in Bakelite valves is vital for the gas industry, considering both gas distribution management and health, safety, and environmental (HSE) concerns [9].

In recent decades, researchers have investigated the effects of irradiation on polymeric materials for various applications. These investigations show that the structural changes induced by radiation in polymers are influenced by the type of irradiation [10] and the specific conditions under which it occurs, such as the surrounding atmosphere [11, 12], the thickness of the samples, and the dosage of irradiation [13,14]. Moreover, these factors can lead to either chain scission or cross-linking through free radical reactions [10-14]. In addition, cross-linking in polymers enhances their mechanical, physical, and chemical properties, including hardness, tensile strength, impact resistance, glass transition temperature (T_g), electrical insulation, and resistance to acidolysis, hydrolysis, and ozonolysis. Conversely, chain scission generally results in a weakening of these properties [15, 16].

Published studies on the effects of irradiation on PF resins have shown similar behavior. Specifically, irradiation may lead to the formation of a three-dimensional structure by interconnecting polymer chains and networks through free radical reactions. Moreover, this process can result in the creation of new covalent bonds, while also can potentially degrade the polymer chains [13,14].

This study investigates the effects of different dosages of 12 MeV electron beam irradiation on the mechanical and physical properties of Bakelite valves used in conventional diaphragm gas meters (see Fig. 1a). These valves are not pure Bakelite; they contain additives that could influence their irradiation response. In addition, this consideration, along with a request from Fars Province Gas Company (FPGC) to explore the reasons behind gas losses in gas meters, prompted our investigation. Surprisingly, we found no prior reports addressing this issue. Consequently, we decided to identify and resolve the problem. In this study, we conduct a thorough analysis of these valves and examine the effects of electron beam irradiation—a single modification process—on the quality of the manufactured valves. The irradiation is performed at room temperature and under vacuum conditions. Furthermore, the resulting changes in the properties of the Bakelite specimens are analyzed using several characterization techniques, including Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), tensile strength tests, impact resistance tests, and swelling assessments. This research aims to provide a better understanding of how electron beam irradiation induces microstructural modifications in the Bakelite valves of gas meters.

Materials and Methods

Materials

Bakelite valves for G4 diaphragm gas meters were supplied by FPGC. Fig. 1b shows the Bakelite valves and valve seats. As indicated in Table 1, these valves were categorized into two distinct types: sanded (smooth, designated as "L," which stands for "Lapped" in this article) and unsanded (plain). Sanding has become an industrial standard to ensure the smoothness of the touching-surfaces of both the valves and their seats, which is crucial for preventing internal leakage. Several square sheets, each with approximate dimensions of 1.0 cm × 1.0 cm × 0.3 cm, were prepared from these valves (see Fig. 1c). The samples, which included the two parts of the valves—the seats and the moving lids (illustrated in Fig. 1b)—as well as the prepared square pieces, were placed in polyethylene bags and vacuum-sealed prior to irradiation.


a.


b.


c

Fig. 1. a A conventional diaphragm gas meter with Bakelite valves, b. the two parts of Bakelite valves (moving lid and the seat) used in the present research, c. the prepared sample for XRD, AFM and SEM.

Table 1 Notations used in the present article

Notation	Description
S0	Unsanded sample and not irradiated
S60	Unsanded sample and irradiated at 60 kGy
S80	Unsanded sample and irradiated at 80 kGy
S0L	Sanded sample and not irradiated
S60L	Sanded sample and irradiated at 60 kGy
S80L	Sanded sample and irradiated at 80 kGy

Electron Beam Irradiation

Multiple Bakelite samples (see Fig.s 1b and 1c) were vacuum-pressed in polyethylene bags. Following vacuum process, the samples were subjected to electron beam irradiation using a Rhodotron-TT200 electron accelerator (Yazd Radiation Application Research Center). There, the samples received a 12 MeV electron beam at room temperature, with doses of 60 and 80 kGy. Finally, the irradiated samples at different doses were then analyzed using a range of characterization techniques.

Characterizations

FTIR was utilized to investigate changes in the chemical nature of the samples due to electron beam irradiation. Moreover, the spectra were recorded using a Bruker instrument (Model: Tensor II). In addition, XRD analyses were conducted to study the variations in crystallinity and composition resulting from irradiation, also employing a Bruker instrument (Model: Advance D8). Furthermore, to examine changes in the morphology and composition of the surface of the samples due to irradiation, SEM/EDX analyses were performed with a TESCAN Vega3 instrument. For assessing the changes in the topology of the surfaces, AFM analysis was carried out using a Naio AFM instrument. Moreover, TGA was used to study alterations in thermal stability and decomposition temperature of the samples due to irradiation, with tests performed on a TGA/DSC 1, star® system at a heating rate of 5 °C/min under a nitrogen atmosphere. In addition, swelling tests of the samples were conducted in toluene and water to determine the amount of solvent uptake by the irradiated and non-irradiated samples according to ASTM D570. Additionally, impact and tensile tests were performed on the samples using Gotech and UTM instruments, according to ASTM D256 and ASTM D638 standards, respectively.

Results and Discussion

In the first stage, the chemical composition of sanded and unsanded samples was analyzed using FTIR. Fig. 2a shows the FTIR spectra of unsanded sample while Fig. 2b shows the spectra for sanded sample. As shown in Fig. 2b, three distinct peaks appear in the spectrum of the sanded sample that are absent or weak in the unsanded sample (Fig. 2a): the peak at 3375 cm^{-1} corresponds to the stretching vibration of hydroxyl groups, while the peaks at 1022 cm^{-1} and 755 cm^{-1} correspond to the bending vibrations of these groups. The appearance of these peaks indicates that sanding changes the chemical structure of Bakelite valves, resulting in the formation of polar hydroxyl groups on their surface. This alteration is harmful to gas meters because these polar groups can adsorb and absorb polar compounds found in the gas stream, such as nitrogen, oxygen, and sulfur-containing compounds. Consequently, the valves may swell and distort, especially when the crosslinking density is uneven across the sample. This inconsistency causes certain areas to swell more than others, leading to twisting and deformation of the components. Ultimately, this results in the misalignment of the seat and the moving lid, which can lead to inaccurate gas volume measurements. This finding serves as an important warning for manufacturers of diaphragm gas meters.

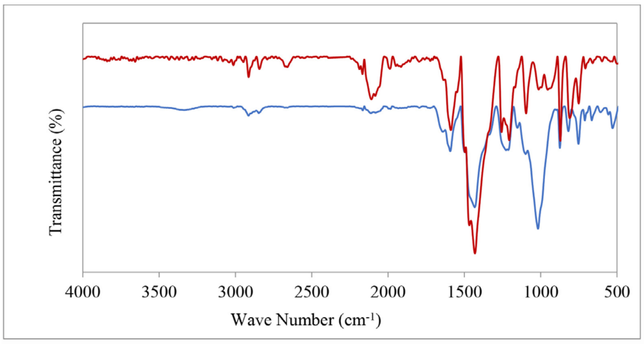

Fig. 2 FTIR analysis of (a). unsanded and (b). sanded Bakelite species.

To investigate the effect of electron beam irradiation on the chemical nature of the valves, the FTIR spectra of the irradiated and non-irradiated sanded samples are compared in Fig. 3.

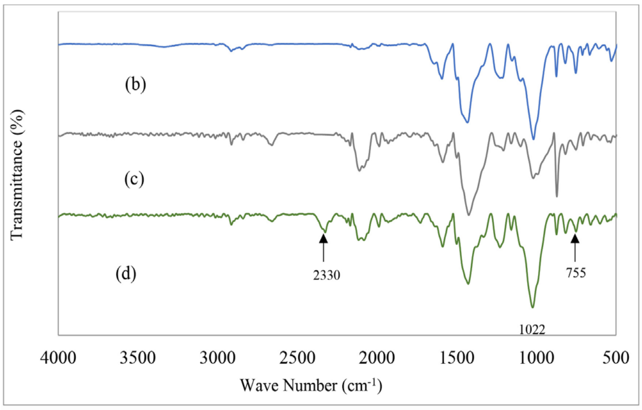

Fig. 3 FTIR spectra of sanded Bakelite species irradiated at b. 0, c.60, d.80 KGy.

It is generally accepted that electron beam irradiation can cause degradation or crosslinking in polymers. As a result of these phenomena, new peaks may appear in spectra, existing peaks may shift in position, or some peaks may disappear. As shown in Fig. 3, electron beam irradiation at either 60 or 80 kGy has completely eliminated the peaks associated with the hydroxyl group. This phenomenon may be attributed to the condensation or cleavage of hydroxyl groups due to the irradiation. Additionally, three new peaks at 1725, 2330, and 2650 cm^{-1} appear in the samples treated at 80 kGy which may originate from new functional groups, particularly formyl (C=O) and C=C groups, which could have formed during degradation processes [13, 15, 17, 18]. For example, the peak appearing at 2330 cm^{-1} may correspond to trapped.

CO_2, which can originate from Bakelite degradation [18].
The XRD patterns of both sanded and unsanded samples without irradiation are presented in Fig. 4. Fig. 5 illustrates the effects of irradiation on these patterns. As shown in the figures, the overall diffraction pattern did not change as a result of irradiation. Moreover, the area under the sharp peaks at 26.5, 29.5, and 54 degrees, as well as the halo between 5 and 19 degrees, was precisely calculated using Origin Pro 2015. In addition, to determine the levels of crystallinity (%X), the area beneath the sharp peaks was divided by the total area, which includes both the sharp peaks and the halo. The resulting levels of crystallinity are detailed in Table 2. The results indicate that the lowest level of crystallinity corresponds to the samples irradiated at 60 kGy. Crosslinking generally decreases the crystallinity of polymers [19, 20]. This is because crosslinks, which are chemical bonds between polymer chains, disrupt the ordered arrangement of molecules needed for crystallinity. Crosslinking can also hinder chain mobility, making it more difficult for chains to align into crystalline structures. Conversely, the higher levels of crystallinity observed at 80 kGy may be attributed to increased degradation of the polymeric chains at that dose. Shorter chains are better able to orient and pack closely together compared to longer or crosslinked chains [21, 22]. Additionally, the increased crosslinking at 60 kGy, in comparison to the non-irradiated sample and the sample irradiated at 80 kGy, likely reduced the ability of the chains to move and orient alongside one another.

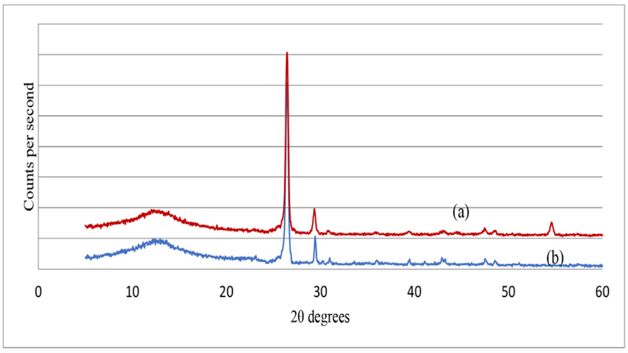

Fig. 4 XRD analysis of (a). Unsanded and (b). sanded Bakelite species.

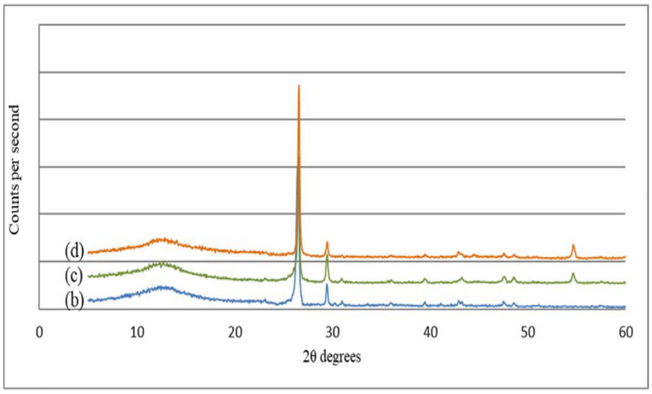

Fig. 5 XRD analysis of sanded samples irradiated at (b).0, (c). 60 and (d) 80 kGy.

Table 2 XRD parameters of the samples.

Sample	S0L	S0	S60L	S60	S80L	S80
of crystalline peaks #	2	2	2	2	2	2
Peak heights	5500	3000	2700	3500	4000	4000
X %	43	37	37	33	40	35

An intriguing finding from this analysis is the relatively high levels of crystallinity observed in all the samples. Typically, Bakelite is recognized as an amorphous polymer, with crystallinities ranging from 16% to 21% [13]. However, this study reveals higher levels of crystallinity, which may be attributed to the influence of additives in the Bakelite samples, such as wood powder, graphite, and calcium carbonate. These additives may enhance nucleation and crystallization in the samples. Another observation is that, in general, the sanded species exhibit higher crystallinities after irradiation. This may be attributed to the breaking of polymer chains during the sanding process. As mentioned previously, shorter chains can more easily orient themselves beside one another. Additionally, the heat generated during sanding can help to anneal and align the chains at the surface which can be considered as one of the reasons behind higher crystallinities of the sanded samples seen in Table 2.
EDX analysis of the samples confirmed the presence of various elements in the analyzed Bakelite samples. Furthermore, the results for one of the samples are reported in Table 3. In addition, sanding or irradiation did not significantly change the compositions.

Table 3 Sample EDX result *

Elt	Line	Int	Error	K	Kr	W%
C	Ka	43.8	2.9356	0.6302	0.2462	55.21
O	Ka	17.7	3.0326	0.1077	0.0421	31.50
Mg	Ka	4.1	10.4305	0.0074	0.0029	0.55
Al	Ka	10.0	10.5873	0.0172	0.0067	1.14
Ca	Ka	65.8	0.8721	0.1866	0.0729	8.22
As	La	12.0	13.7243	0.0510	0.0199	3.38
Total	-	-	-	1.0000	0.3907	100.00

*. Elt: Element, Int: Integral, K: Kα X-ray line, Kr: Kβ X-ray line

Fig. 6 presents the SEM micrographs of the samples. A comparison of the surface morphology shows that the sanded samples (shown in the right column of the figure) generally exhibit a higher number of cracks and crevices. Moreover, this indicates that sanding has adverse effects on their surfaces. These defects can trap potential impurities present in gas streams, which may alter the structure of the valves over long-term use.

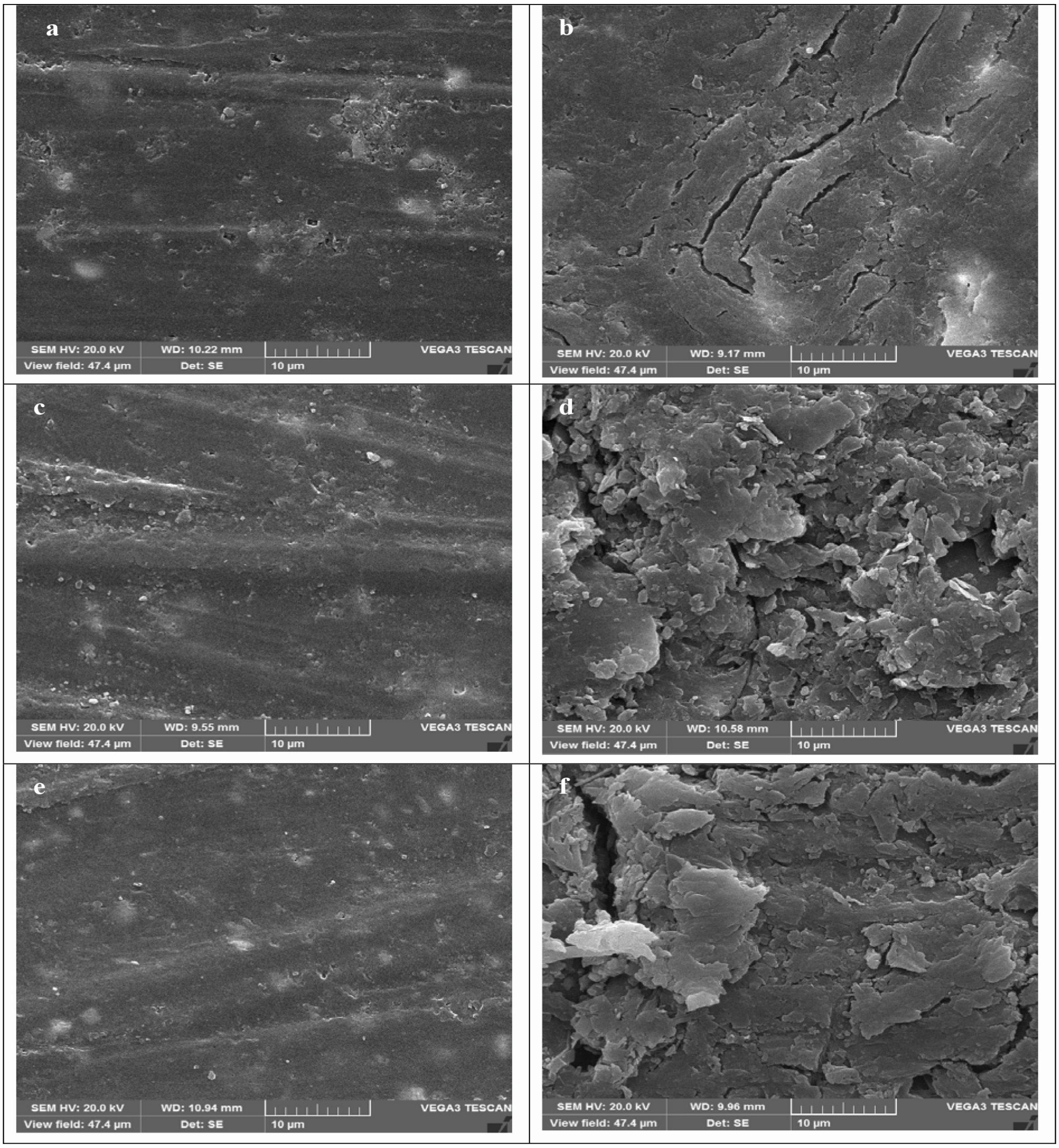

Fig. 6 SEM micrographs of the samples; unsanded and irradiated at a. 0 kGy, c. 60 kGy, e. 80 kGy; sanded and irradiated at b. 0 kGy, d. 60 kGy, f. 80 kGy. The magnification of all of the graphs 4kx.

Additionally, when comparing the surface morphology of the irradiated sanded samples, it is evident that those irradiated at 80 kGy exhibit the poorest surface quality, suggesting significant degradation at that dosage. In contrast, the sample treated at 60 kGy shows a more satisfactory surface morphology.

The AFM topographic images of the samples are presented in Fig. 7, while the corresponding roughness parameters are detailed in Table 4. After irradiation, the topology of the samples changed, with more notable alterations observed in the S60 samples, resulting in smoother surfaces.

The data in Table 4 indicate that irradiation significantly reduced all four roughness parameters, particularly in the S60 samples, which it suggests effective crosslinking and the achievement of smoother surfaces. Generally, crosslinking can alter the topology of polymers by creating a three-dimensional network structure. In addition, this process restricts the movement of polymer chains, and therefore the mobility and aggregation of the chains is suppressed, leading to a more uniform and smoother surface (lower roughness). Also, it is noted that, the roughness parameters are decreased when the samples treated at 80 kGy but the amount of decrease is less than 60 kGy. This can be indicative of starting the polymer degradation process at 80 kGy [23].

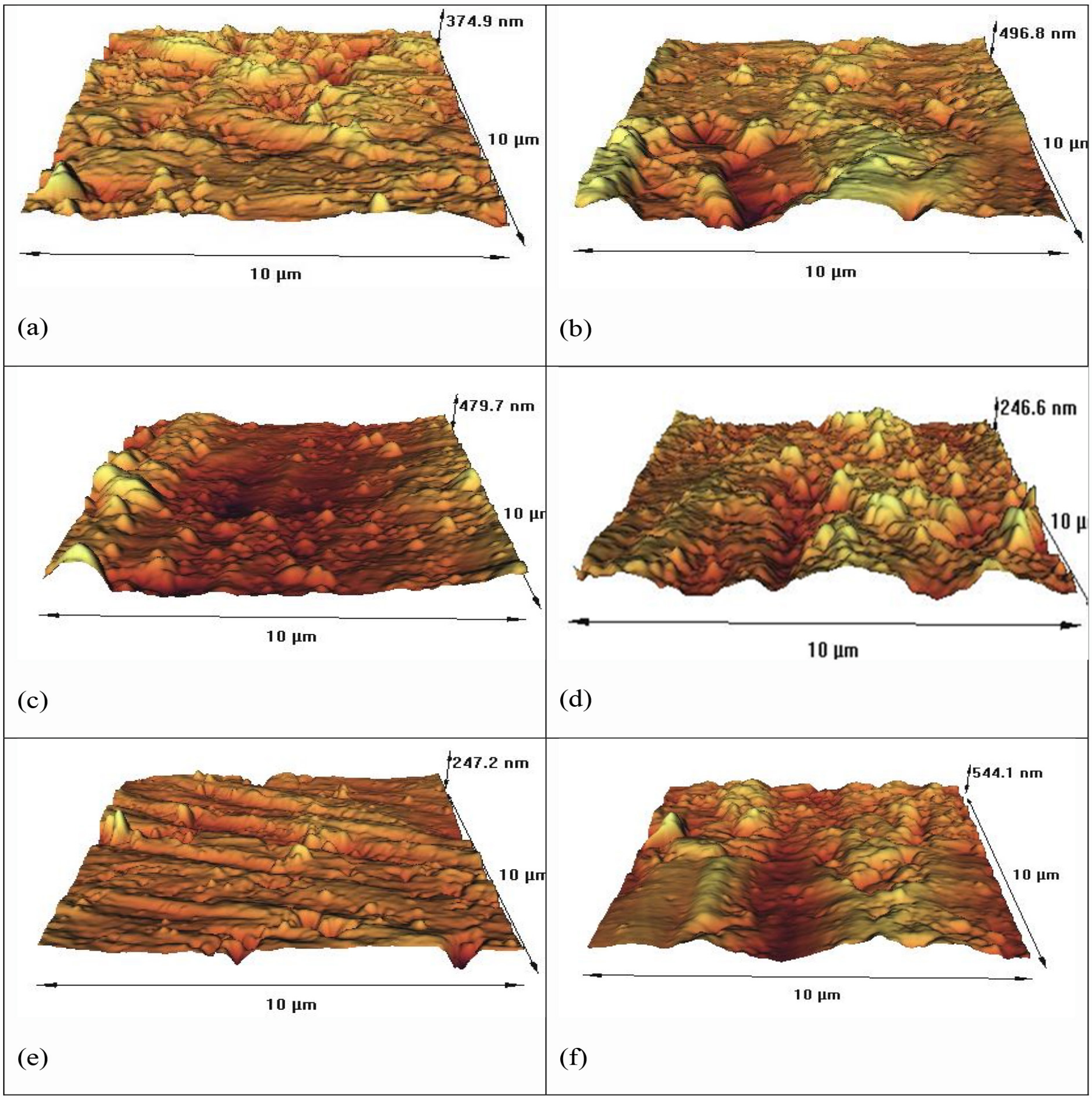

Fig. 7 AFM topographic images of the samples; unsanded and irradiated at 0kGy (a), 60 kGy (c), 80 kGy (e); sanded and irradiated at 0 kGy (b), 60 kGy (d), 80 kGy (f).

Table 4 Effect of electron beam irradiation on the Roughness Parameters of samples.

Entry	Arithmetic mean deviation (average roughness) S_a or R_a (nm)	Root means square deviation (RMS) S_q or R_q (nm)	Arithmetic averageHeight S_z or R_z (nm)	average roughness Mean (nm)	SD (nm)
S0	3.172	24.37	74.10	2.2077E-7	37.93
S0L	16.81	128.0	109.5	1.3422E-6	63.943
S60	2.431	18.35	79.99	5.9837E-7	71.16
S60L	1.890	13.36	73.17	5.9837E-7	71.16
S80	4.032	32.26	41.44	3.4189E-7	17.66
S80L	5.442	42.50	164.4	1.6723E-6	69.642

Fig. 8 and Table 5 present the TGA/DTG results for sanded samples before and after irradiation. The results indicate that irradiation at 60 kGy resulted in a shift of the degradation and decomposition temperatures (MRDT and IDT) to higher values. In contrast, at 80 kGy, the changes in these temperatures were less pronounced. In addition, the degradation and decomposition amounts (1st and 2nd stages) of the S60L sample were lower than those of the S0L and S80L samples, suggesting that 60 kGy is an optimal dosage for enhancing the thermal stability of this specific type of Bakelite sample. Furthermore, the maximum rate of decomposition (MRD) for the samples irradiated at 60 kGy was the lowest, demonstrating improved thermal stability. In addition, this enhanced thermal stability may be attributed to the cross-linking of samples at 60 kGy.

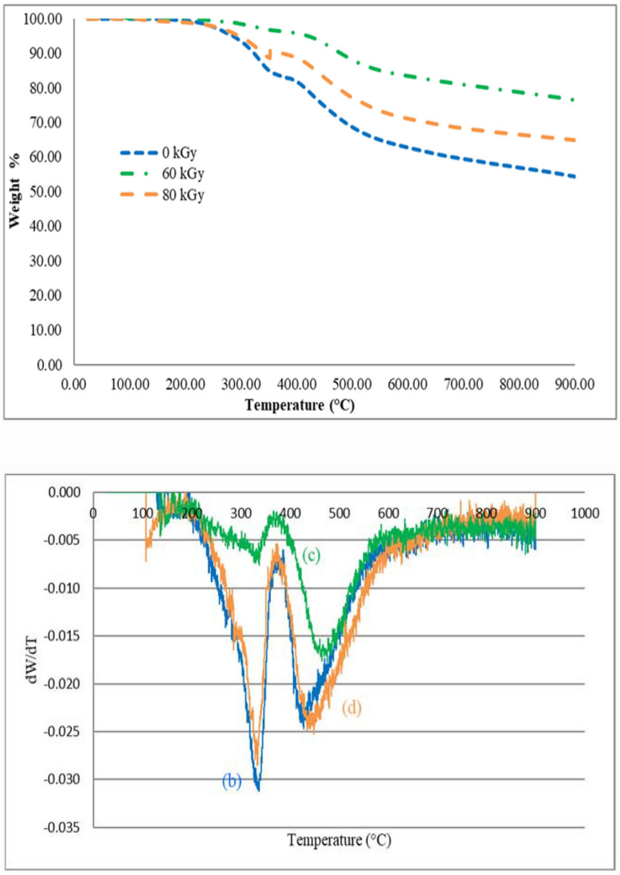

Fig. 8 TGA and DTG curves of sanded samples irradiated at (b).0, (c).60, (d) 80 kGy.

Irradiation at 60 kGy creates new crosslinks in the sample, making it more resistant to degradation. As a result, the thermal stability of the sample increases. However, at 80 kGy, degradation begins to occur, causing some bonds to break, and lower improvements in thermal stability are observed for the sample.

Another physical test conducted on the samples involved examining their swelling behavior in two different media: water and toluene. Furthermore, Table 6 presents the swelling amounts for the samples in both solvents. At first glance, it is evident that the swelling is considerably higher for all samples compared to typical values seen for phenolic resins [24]. However, when considering the composition of the valves, it becomes clear that they contain various types of fillers (including wood powder, graphite, calcium carbonate, etc.) that contribute to water uptake. Among these fillers, wood powder, added to the formulations, can significantly affect water absorption. Additionally, the results show that all sanded samples exhibited higher water uptake than unsanded samples. This indicates that while sanding is crucial for ensuring a tight fit in the valves (specifically at the lids and seats), it inadvertently causes an increase in water absorption, which can lead to swelling and twisting of the parts. In addition, this higher water uptake of the sanded samples can be due to the presence of higher amounts of polar groups presented on the surface after sanding in air. Moreover, the results indicate that irradiating the samples at both 60 kGy and 80 kGy did not significantly alter the degree of swelling in water.

Table 5 TGA/DTG results of sanded sample irradiated at different doses.

Entry	1st IDT (°C)	Amount of 1st decomp. (%)	1st MRDT (°C)	1st MRD (mg/°C)	2nd IDT (°C)	Amount of 2nd decomp. (%)	2nd MRDT (°C)	2nd MRD (mg/°C)
S0L	160	13.56	329	0.030	370	17.73	427	0.025
S60L	192	4.0	330	0.008	380	13.5	462	0.020
S80L	110	6.88	329	0.030	350	15.81	440	0.025

IDT: Initial Decomposition Temperature
MRDT: Maximum Rate Decomposition Temperature
MRD: Maximum Rate of Decomposition

Table 6 Swelling results of the samples.

Sample	Solvent	S0	S0L	S60	S60L	S80	S80L
Swelling %	Toluene	0	0	0	0	0	0
Swelling %	Water	0.4	0.5	0.6	0.7	0.4	0.5

However, a slight increase in swelling was observed at 60 kGy. Crosslinking typically reduces the swelling of polymeric compounds. More crosslinks mean a denser network, restricting the polymer chains from expanding and absorbing more solvent. Moreover, crosslink points hold the chains together which resist toward swelling. If the number of the mentioned points increases, the resistance against swelling goes up. By comparing the swelling data obtained for irradiated samples at 60 and 80 kGy, the lower amounts of swelling obtained for the samples treated at 80 kGy can be considered as a clue for the lower amounts of crosslinking in the sample which can be indicative of prevalence of degradation over crosslinking at this dose. This finding is supported by other findings in previous sections particularly those from FTIR, TGA, and SEM analyses. Furthermore, no swelling was observed in toluene under any conditions, regardless of whether the samples were irradiated, due to the mismatch in polarity between toluene and Bakelite. Mechanical testing of the sanded samples included assessments of impact strength and tensile strength. The results are summarized in Table 7. The table shows that the maximum values were achieved for the sanded sample irradiated at 60 kGy, indicating effective crosslinking between the polymer chains. In contrast, at 80 kGy, degradation predominated, resulting in a decline in the mechanical properties.

Table 7 Tensile strength and impact strength test results.

Sample	S0L	S60L	S80L
Impact strength ($kJ.m^{-2}$)	38	106	53
Tensile Strength (MPa)	16.2	16.5	10.7

Conclusions

The results demonstrated that upon electron beam irradiation of Bakelite valve samples, crosslinking and decomposition occur simultaneously. At an irradiation dose of 60 kGy, crosslinking dominated over degradation and the physical and mechanical properties of the samples improved. However, at 80 kGy, degradation dominated and either deterioration or minimal changes of properties were observed. Notably, irradiation at 60 kGy reduced average roughness by 55% and increased impact strength by 180%. In conclusion, electron beam irradiation under vacuum can result in favorable modifications in Bakelite components used in diaphragm gas meters, potentially addressing issues of unaccounted gas and related economic losses.

Nomenclatures

IDT: Initial Decomposition Temperature
MRDT: Maximum Rate Decomposition Temperature
MRD: Maximum Rate of Decomposition

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Conflicts of Interest or Competing Interests

We are not aware of any potential conflicts of interest.

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Graphical Abstract