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Preparation and Application of Radiation-crosslinked Polypropylene by One-step Process for Corrosion Prevention of High-temperature Pipelines

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Abstract: *Using propylene-ethylene block copolymer (PPB) and metallocene polypropylene random (mPPR) as matrix, olefin block copolymer (OBC) and metallocene polyethylene (mPE) as reinforcement, trimethylolpropane trimethacrylate as sensitizer, antioxidant 1010 and dioctadecyl 3,3-thio-di-propionate as antioxidant, the radiation-crosslinked polypropylene with excellent high-temperature performance was prepared by one-step process and it was used to produce the heat shrinkable tape with three layers structure further. The effect of the formula, the orientation, the crosslinking, the heat treatment and shrink on the crosslinked-polypropylene were studied. The results show that the crosslinked-polypropylene has optimum overall property when the amounts of PPB, mPPR, OBC, and mPE were 50, 10, 15, and 15 phr, respectively. After the orientation in one-step process and the radiation crosslinking with the irradiation dose of 6 Mrad, the tensile strength, the elongation at break, the shrinkage ration, the gel content of the crosslinked-polypropylene reached 33 MPa, 390%, 30%, 51%, respectively. After heat treatment, its mechanical properties further improved and the decrease of its tensile strength and elongation at break did not exceed 15% after thermal ageing at 130 ^{o}C for 100 days. Moreover, the corresponding heat shrinkable tapes show outstanding peel strength, impact strength and cathodic disbondment properties in various conditions.*

Keywords: *polypropylene, radiation crosslinking, one-step process, high-temperature performance, heat shrinkable tape*

1. Introduction

Pipeline transportation is the major mode for long-distance transportation of oil and natural gas due to its large transport capacity, high stability and economic advantage [1]. With the depth development of oil and gas resources in deep sea areas, the safety of the submarine pipelines is paid great attention to avoid any leakage accident resulting in immeasurable economic losses and environmental pollution [2]. Significantly, corrosion is the main forms of the destruction of submarine pipelines by statistics analysis on the relevant leakage accidents [3, 4]. At present, the corrosion prevention of submarine pipelines relies chiefly on polymer matrix anticorrosive coating such as polyethylene anticorrosive coating and polypropylene anticorrosive coating [5, 6].

High-temperature operating condition is a grand challenge for the anticorrosive coating to provide effective and long-term protection for pipeline. At the condition below critical temperature, the wax crystals will precipitate during the pipeline transportation of waxy oil, leading to pipe blockage and even pipe burst [7]. And the pipeline transportation of natural gas also experiences the same situation, due to the water which is difficult to remove completely and will produce the precipitate of ice crystals at low temperature [8].

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Therefore, transportation media should be carried out by temperature-rise treatment during pipeline transportation, especially submarine pipelines at low temperature environment all along [9]. Furthermore, the weld seam is the weakest part of the corrosion prevention of submarine pipelines, and has vital effect on the stable operation of the submarine pipelines during their designed service life. And the corrosion prevention at weld seam sets higher demands on the performances of the anticorrosive coating as well. Compared with conventional anticorrosive coating of pipeline transportation, poly-propylene anticorrosive coating shows preferable high-temperature performance [10], and further it is also more suitable for preparing the crosslinked-polypropylene heat shrinkable tape which is used for the corrosion prevention at weld seam.

Crosslinking modification, an effective method for expanding the application of polypropylene [11], can enhance its mechanical properties, heat-resistant quality, resistance to oxidative degradation, and low temperature brittleness, mainly including organic peroxide crosslinking [12], silane crosslinking [13], and radiation crosslinking [14]. Radiation crosslinking is considered as suitable for industrial production owing to its simple craft, controlled process, and environmental advantage. However, the degradation reaction and the crosslinking reaction are simultaneous in the radiation crosslinking process of polypropylene, causing low crosslinking efficiency [15]. In recent years, various research have been carried out in order to increase the degree of crosslinking, such as the control of irradiation dose and irradiation dose rate, the selection of irradiation atmosphere, and the addition of sensitizer. First, the concentration of free radical will increase with the increase of irradiation dose and irradiation dose rate, improving the probabilities of degradation reaction and crosslinking reaction simultaneously. By controlling irradiation dose and irradiation dose rate, the appropriate concentration of free radical can be obtained which is conducive to crosslinking reaction rather than degradation reaction or branching reaction [16, 17]. Besides, the tertiary carbon radicals are produced during the irradiation of polypropylene and the high activity causes them to react easily with oxygen to produce the alcoxyl radicals. The alcoxyl radicals are beneficial to degradation reaction and can restrain crosslinking reaction, so isolating oxygen and inert-gas protection can improve the crosslinking efficiency significantly [18, 19]. In addition, the double carbon bonds in sensitizer, serve as bridged linkage, can link diverse free radical produced by polypropylene to form cross-linked structure or branched structure. Therefore, the addition of sensitizer contribute to increase the degree of crosslinking but an excess of addition will lead to losing efficacy due to the self-crosslinking of sensitizer [20, 21].

On the other hand, the enhancement of crystallization by orientation is a feasible way to improve the performances of polypropylene anticorrosive coating, particularly mechanical property, high-temperature thermal stability, and heat aging resistance [22, 23]. Currently in the industrial production, the stretching orientation process with thermal treatment, as an additional process, is used to enhance the crystallization after the extrusion molding process with radiation crosslinking, bringing many extra losses including the energy consumption, the materials loss during side cutting, and the reduction of efficiency. Obviously, it is a critical factor for the corrosion prevention of submarine pipelines how to exploit a one-step process combining extrusion molding and orientation to take the place of the two-step process.

In this work, the radiation-crosslinked polypropylene with enhanced high-temperature performance was prepared by one-step process to deal with the high-temperature operating condition and provide long-term protection for pipeline, using various polypropylene, polyethylene and corresponding co-polymer to build the polypropylene composite, and further it was used to produce the heat shrinkable tape with three layers structure. Benefitting by the orientation in the extrusion molding process and the contribution of each component in the matrix formula to the mechanical properties, the crosslinking reaction, and the high-temperature performance, the radiation-crosslinked polypropylene and the corresponding heat shrinkable tape show excellent application prospect in the corrosion prevention field of high-temperature pipelines.

2. Materials and methods

2.1. Materials

Propylene-ethylene block copolymer (PPB) was provided by Sinopec Yanshan Petrochemical Company. Metallocene polypropylene random (mPPR) was obtained from Japan Polypropylene Corporation. Ethylene-octene block copolymer (Olefin block copolymer, OBC) was supplied by DowDuPont Company. Metallocene polyethylene (mPE) was bought from Prime Polymer Corporation. Trimethylolpropane trimethacrylate (TMPTMA) was offered by Eternal Chemical Co. Ltd. Pentaerythritol tetrakis (3-(3, 5-di-tert-butyl-4-hydroxyphenyl) propionate) (antioxidant 1010) and dioctadecyl 3,3-Thiodipropionate (DSTDP) were purchased from BASF SE.

2.2. Preparation of the crosslinked-polypropylene

PPB, mPPR, OBC, mPE, TMPTMA (sensitizer), and the antioxidants were mixed uniformly in a high-speed mixer (JSY-L200KG, Dongguan Jiesiya Mechanical Equipment Co. Ltd) according to the formula. The blends were extruded by a twin-screw extruder (Model 65, China BlueStar Chengrand Research Institute of Chemical Industry) at 200°C, and then went through cooling, air drying, cutting. After dring at 90°C, the prepared granules of the blends were extruded and oriented with a one-step process by a single-screw extruder (Model 90, Shanghai Jinwei Extrusion foaming Technology Co. Ltd), and then the PP sheets were obtained (as shown in Figure 1 (a)). The relevant temperatures at each stage in the single-screw extruder were 100°C, 120°C, 150°C, 180°C, 190°C, 200°C, 210°C, 210°C, respectively. Next, the PP sheets were modified in the radiation-crosslinked process with various irradiation doses by an electron accelcrator (Model ELV, Jiangsu Dasheng Accelerator Manufacturing Co. Ltd), and the radiation-crosslinked polypropylene sheets were prepared.

2.3. Preparation and installation of the crosslinked-polypropylene heat shrinkable tape

The homemade polypropylene hot-melt adhesive with a thickness of 1.2 mm was coated on the surface of the crosslinked-polypropylene sheet with a thickness of 1.0 mm by a twin-screw extrusion coating equipment, and the crosslinked-polypropylene heat shrinkable tape was obtained after cooling. And the polypropylene hot-melt adhesive was the mixture of maleic anhydride grafted propylene-ethylene block copolymer (PPB-g-MAH), maleic anhydride grafted polypropylene random (PPR-g-MAH), maleic anhydride grafted polyolefin elastomer (POE-g-MAH), hydrogenated C_5 petroleum resin, graphene oxide, and 4,4-Thiobis (6-tert-butyl-3-methylphenol). Then the crosslinked-polypropylene heat shrinkable tapes were bonded on the steel painted the epoxy primer and the polypropylene anticorrosive coating at weld seam (Figure 1b), and the installation was completed after being baked by open fire.

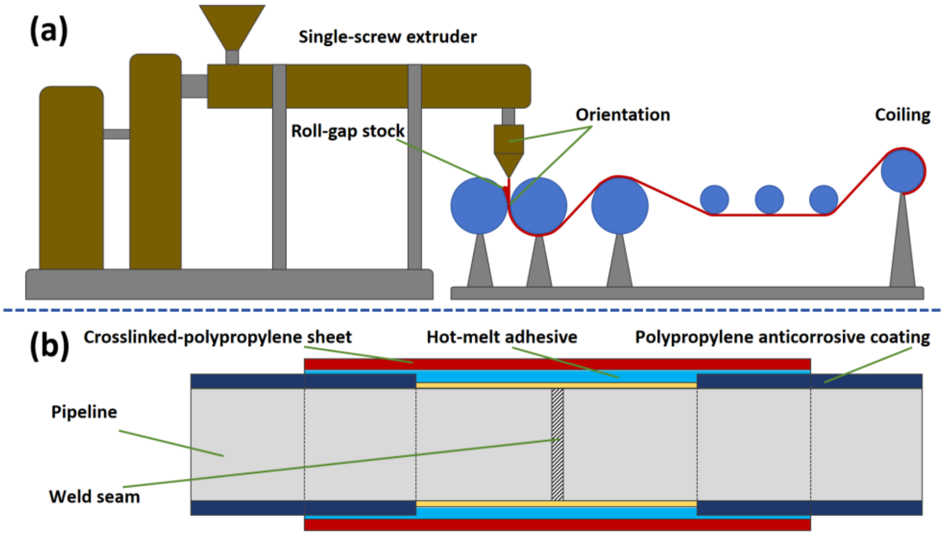

Figure 1. Schematic diagram of (a) the one-step process combining extrusion and orientation of the PP sheets and (b) the crosslinked-polypropylene heat shrinkable tape at at weld seam

2.4. Characterization

The chemical structures and the crystal structures of the crosslinked-polypropylene were determined by a Fourier transform infrared spectroscopy (FITR, Nicolet 6700, Thermo Fisher Scientific Inc) an X-ray diffractrometry (XRD, D8 Advance, Bruker Corporation) respectively. After brittle failure in liquid nitrogen, the fracture morphology of the crosslinked-polypropylene sheets was observed by a scanning electron microscopy (SEM, JSM-5510LV, JEOL). The mechanical properties were measured by an electronic universal tensile testing machine (CMT4203, MTS Systems Corporation) according to GB/T1040.2-2023 with a tensile speed of 50 mm/min. After boiling in xylene for 8 h, the sample was washed by ethyl alcohol, dried, and weighed, and then its gel content was calculated according to GB/T18474-2001.

The colligation score of the crosslinked-polypropylene reflects the degree of appropriateness to prepare the heat shrinkable tape, and it is sum of four scores including the tensile strength (TS), elongation at break (EB), melt flow index (MFI), Vicat softening temperature (VST). Both of the full TS score and the full EB score is 30, and both of the TS score of 28 MPa and the EB score of 550% is 24. The tensile strength should be not less than 28 MPa, and the TS score adds 1 or deducts 5 with the tensile strength increase or decrease 1 MPa. The elongation at break should be not less than 550%, and the EB score adds 1 or deducts 6 with the tensile strength increase or decrease 50%. The MFI score is 20 when the melt flow index is in the range of 0.35-2.0 g/10 min at 230^{o}C, and the MFI score deducts 1 with each 10% exceeding. The full VST score is 20 and the VST score of 120^{o}C is 12. The vicat softening temperature should be not less than 120^{o}C, and the VST score adds 0.5 or deducts 2 with the tensile strength increase or decrease 1^{o}C.

The peel strength, the impact strength, and the cathodic disbondment of the crosslinked-polypropylene heat shrinkable tape were measured according to ISO 21809-3, EN 12068, and ISO 21809-3, respectively. The crosslinked-polypropylene heat shrinkable tapes were bonded on the steel painted the epoxy primer and stood for 24 h at room temperature. Then the peel strength of heat shrinkable tape and the steel, the impact strength, and the cathodic disbondment were tested respectively, and the tensile speed of peel measurements is 10 mm/min. Analogously, the crosslinked-polypropylene heat shrinkable tapes were bonded on the polypropylene pipe to test the peel strength of the heat shrinkable tape and the polypropylene anticorrosive coating.

3. Result and discussions

3.1. Effect of formula on the crosslinked-polypropylene

The formula of the prepared crosslinked-polypropylene is critical to the improvement of the mechanical properties and the effective orientation during the one-step process. The PPB and the mPPR were combined to use as the matrix in order to increase the impact resistance and the low temperature resistance, and the ethylene contained in the PPB and the mPPR also could accelerate the crosslinking reaction. Besides, the OBC elastomer was added as flexibilizer, considering that its octylene segment could increase the impact resistance and the low temperature resistance and its ethylene segment was beneficial to enhance the crosslinking density of the polypropylene composite after radiation process. And mPE, a kind of polymer that cross-linked by irradiation more easily than polypropylene, was added to further improve the crosslinking density, and also was conducive to the compatibility of the PPB, the mPPR and the OBC.

In addition, the sensitizer and the antioxidant have significant effect on the performance of the crosslinked-polypropylene. The TMPTMA was added to increase the crosslinking density effectively in lower irradiation dose, preventing an excess of degradation reaction in high irradiation dose. In consideration of high-temperature operating condition and the open fire cured process in the installation of the crosslinked-polypropylene heat shrinkable tape, antioxidant 1010 was added as primary antioxidant to eliminate the free radicals generated in the processing, the installation or the use procedure, and the DSTDP was added as auxiliary antioxidant to decompose the hydroperoxides.

The optimum ratio of polymer matrix was studied by orthogonal experiment and the result was shown in Table 1. The result shows that the amount of mPE has the greatest influence on the mechanical properties of the crosslinked-polypropylene, due to its contribution to the crosslinking reaction and the matrix compatibility. And the order is mPE, PPB, OBC, and mPPR after being sorted by the effect of the amount on the mechanical properties of the crosslinked-polypropylene. The crosslinked-polypropylene with optimum colligation score was obtained when the amounts of PPB, mPPR, OBC, and mPE were 50, 10, 15, and 15 phr, respectively. And the matrix according to this ratio was used to prepare the samples in follow-up experiment.

Table 1. The orthogonal experiment results of the matrix formula of the crosslinked-polypropylene

Serial number	Amount (phr)				Colligation score
	PPB	mPPR	OBC	mPE	
1	45	5	5	5	60
2	45	10	10	10	70
3	45	15	15	15	80
4	50	5	10	15	85
5	50	10	15	5	75
6	50	15	5	10	80
7	55	5	15	10	80
8	55	10	5	15	85
9	55	15	10	5	65
K_1	70.00	75.00	75.00	66.67	
K_2	80.00	76.67	73.33	76.67	
K_3	76.67	75.00	78.33	83.33	
R	10.00	1.67	5.00	16.66	

3.2. Effects of orientation and crosslinking on the crosslinked-polypropylene

The effects of the irradiation dose on the FT-IR spectra and the XRD patterns of the crosslinked-polypropylene were shown in Figure 2. From Figure 2a, the peaks of C=O absorption at 1740 cm^{-1} increases obviously with the increase of the irradiation dose, illustrating more crosslinking reaction due to more irradiation dose. In the radiation crosslinking process, the radiation energy gives rise to the chain scission of polymer and the generation of free radical, and then the free radical leads to the occurrence of crosslinking reaction and the formation of three-dimensional network structure. Therefore, the increase of the irradiation dose is beneficial to the crosslinking reaction, which can increase the crosslinking density of the polypropylene effectively. However, the crystallization peak intensity of the crosslinked-polypropylene at XRD curve decreases and its degree of crystallinity reduces with the increase of the irradiation dose, demonstrating that excess irradiation will destruct the crystal structure (Figure 2b). Obviously, an appropriate irradiation dose to balance the crosslinking density and the degree of crystallinity is significant, in order to obtain the crosslinked-polypropylene with optimum performance.

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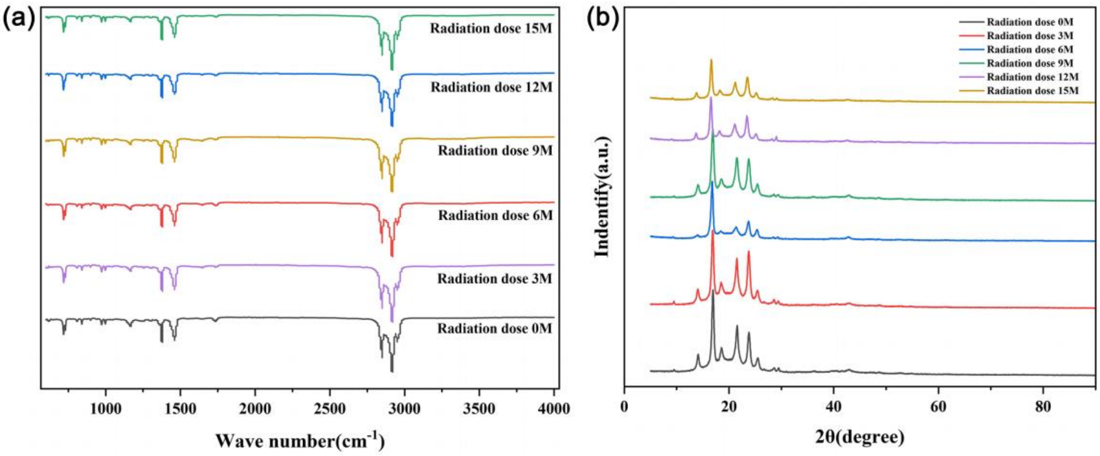

Figure 2. The FT-IR spectra (a) and the XRD patterns (b) of the crosslinked-polypropylene

Figure 3 shows the microtopography of the polypropylene sheet prepared by the one-step process, and the polypropylene sheet without oriented structure prepared by the compression moulding forming was taken as a control. Compared with the control sample, the oriented structure can be observed obviously in the SEM image of the polypropylene sheet prepared by the one-step process. Furthermore, the crystalline structure and cross-linked structure are more ordered along the orientation, and the fracture cracks also tend to extend along the orientation. It is proved that the one-step process can effectively carry out the orientation of the polypropylene sheet and is conducive to the enhancement of its crystallization and the improvement of the performances. Figure 4 shows the effect of the irradiation dose on the microtopography of the crosslinked-polypropylene sheet. With the increase of the irradiation dose, the defects on the fracture surface decrease, and the combination between components of the polypropylene becomes tight due to the crosslinking reaction. However, many interface defects starts to appear after the irradiation dose exceeds 6 Mrad because of the excess irradiation which destructs the crystal structure.

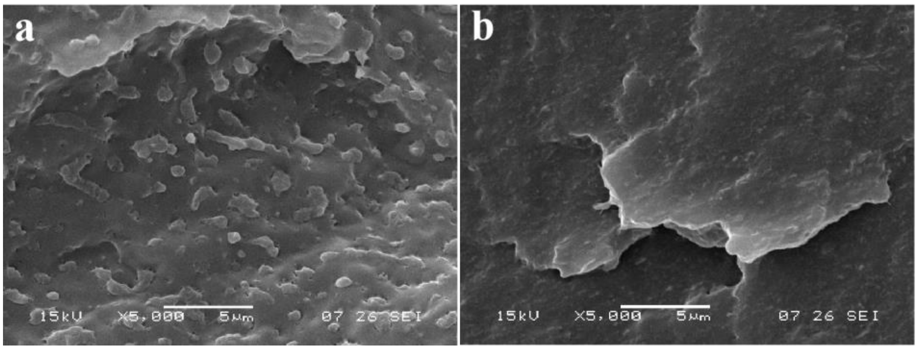

Figure 3. The SEM images of the polypropylene sheets prepared by (a) the compression moulding forming and (b) the one-step process

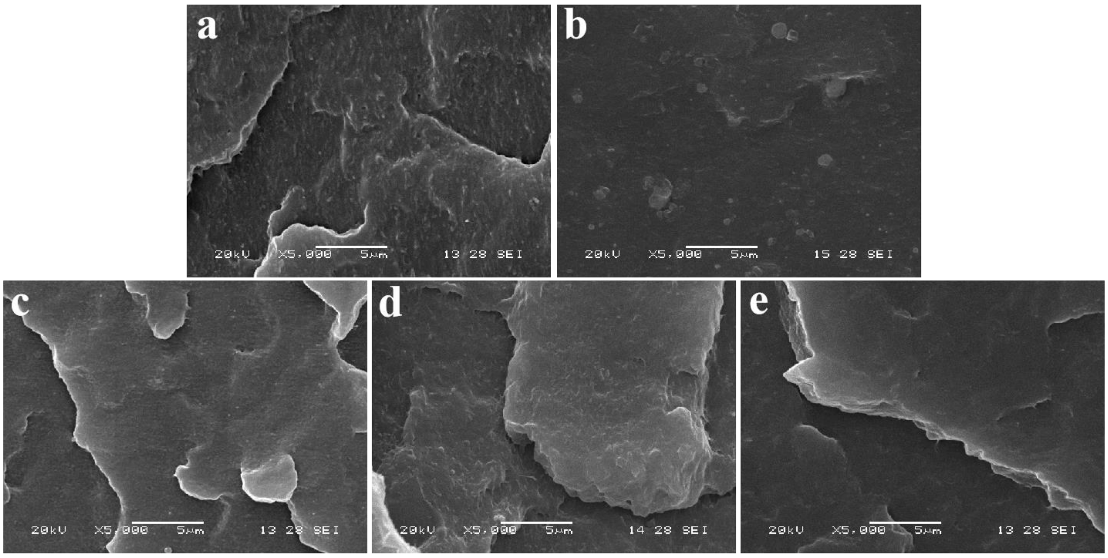

Figure 4. The SEM images of the crosslinked-polypropylene sheets with the irradiation dose of (a) 3 Mrad, (b) 6 Mrad, (c) 9 Mrad, (d) 12 Mrad, and (e) 15 Mrad

The effects of the irradiation dose on the tensile strength, the elongation at break, the shrinkage ration, and the gel content of the crosslinked-polypropylene were shown in Figure 5. With the increase of the irradiation dose, the tensile strength, the elongation at break and the gel content increase, and the shrinkage ration decrease gradually, due to the increase of the crosslinking density. However, the tensile strength and the elongation at break of the crosslinked-polypropylene sheet starts to decline after the irradiation dose exceeds 6 Mrad, because excess irradiation resulted in the destruction of the crystal structure and damaged its mechanical strength. After comprehensive consideration, 6 Mrad is regarded as the appropriate irradiation dose, and the tensile strength, the elongation at break, the shrinkage ration, the gel content of the crosslinked-polypropylene sheets reach 33 MPa, 390%, 30%, 51%, respectively. Compared with the uncrosslinked polypropylene, the shrinkage ration of the crosslinked-polypropylene decrease by about 54%, and the tensile strength, the elongation at break, the gel content of the crosslinked-polypropylene increase by about 14%, 22%, 1200%, respectively.

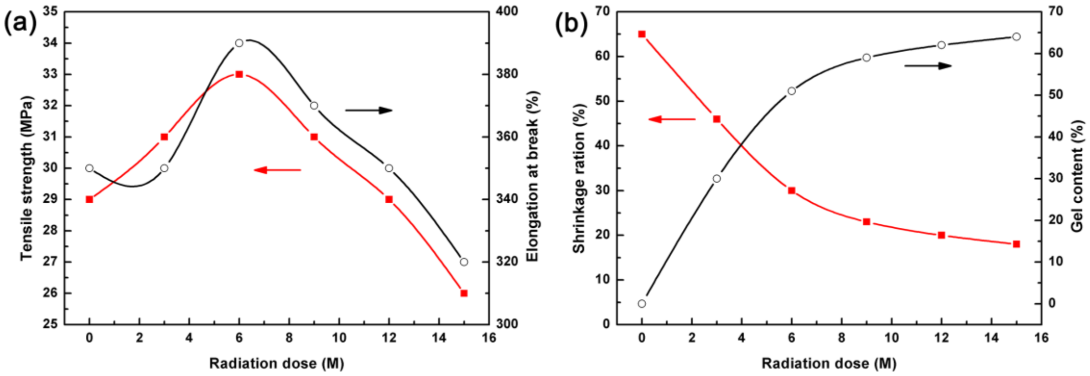

Figure 5. The mechanical properties of the crosslinked-polypropylene sheets

3.3. Effects of heat treatment and shrink on the crosslinked-polypropylene

During the installation process of the crosslinked-polypropylene heat shrinkable tape for the corrosion prevention at weld seam sets, the crosslinked-polypropylene matrix need to be baked by open fire so that the matrix shrinks and is beneficial for the seal at weld seam sets. In addition, through the heat transfer of matrix, the polypropylene hot-melt adhesive will melt and form the tight adhesion with the epoxy primer and the polypropylene anticorrosive coating of the lap joint to effectively enhance the corrosion prevention. Therefore, the heat treatment and free shrinkage at 200°C for 5 min were used to simulate the baking process in open fire during the installation process.

The effect of the irradiation dose on the microtopography of the crosslinked-polypropylene sheet after the heat treatment and free shrinkage was shown in Figure 6. With the increase of the irradiation dose, the defects on the fracture surface first increased and then decreased similarly, and the combination between components of the polypropylene was most stable with the irradiation dose of 6 Mrad because of the excess irradiation which destructs the crystal structure. Besides, compared with the samples before the heat treatment (Figure 5), the fracture surface of the samples after the heat treatment and free shrinkage became rougher obviously, demonstrating that the molecular chains of the crosslinked-polypropylene had indeed shrinked.

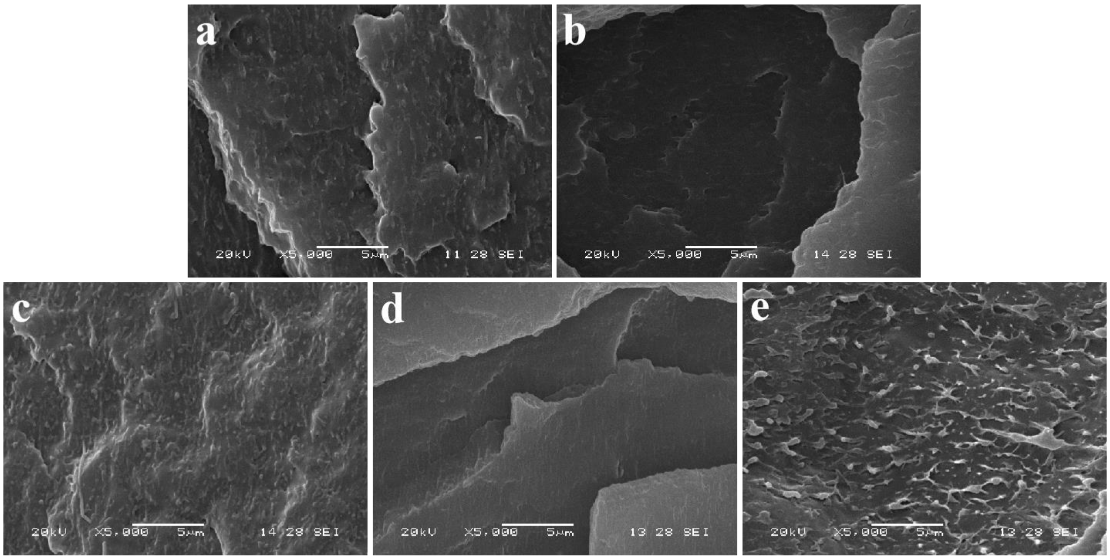

Figure 6. The SEM images of the crosslinked-polypropylene sheets with the irradiation dose of (a) 3 Mrad, (b) 6 Mrad, (c) 9 Mrad, (d) 12 Mrad, and (e) 15 Mrad after the heat treatment and free shrinkage

The effects of the heat treatment and free shrinkage on the mechanical properties of the crosslinked-polypropylene were shown in Figure 7a,b. Compared with the samples without heat treatment, the tensile strength of the samples after heat treatment enhanced slightly and the elongation at break improved observably, benefitting to minishing the weld seam sets and enhancing the corrosion prevention. The shrinkage of the molecular chains increased the degree of entanglement and the curl chains could spread renewedly to enhance the mechanical properties of the crosslinked-polypropylene. Furthermore, the high temperature resistance of the crosslinked-polypropylene is pivotal to long-termly protect the pipelines with high temperature. As shown in Figure 7c, its stress-strain curve has no significant change after thermal ageing at 130°C for 100 days, and the decrease of its tensile strength and elongation at break did not exceed 15%. Compare with the uncrosslinked polypropylene which was serious destructed and become the state of fragmentation after thermal ageing at 130°C for 100 days, the radiation-crosslinked polypropylene have excellent heat aging resistance. It was proved that the crosslinked-polypropylene had excellent high temperature resistance due to the orientation in the extrusion molding process and the contribution of each component in the matrix formula.

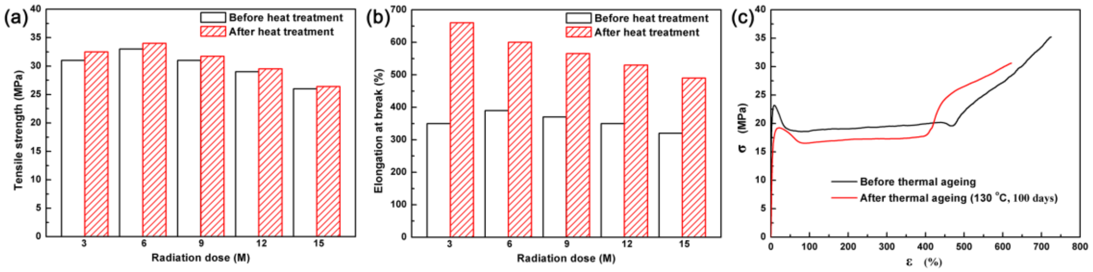

Figure 7. The mechanical properties (a, b) and the thermal ageing resistance (c) of the crosslinked-polypropylene sheets after the heat treatment and free shrinkage

3.4. Properties of the crosslinked-polypropylene heat shrinkable tape

The prepared crosslinked-polypropylene was used to produce the heat shrinkable tape, and the properties of the crosslinked-polypropylene heat shrinkable tape were shown in Table 2. Its peel strengths with both the steel painted the epoxy primer and the polypropylene anticorrosive coating are excellent, and outdistance the standard of commercial heat shrinkable tapes (peel strength ≥ 6 N/mm at 23°C and ≥2 N/mm at 110°C). It reflects that the interfaces between the heat shrinkable tape and all part of the pipelines are stable at both room temperature and high temperature. And the impact strength exceeds 10 J/mm, preventing the impact failure in the installation process of the crosslinked-polypropylene heat shrinkable tape. Moveover, the cathodic disbondment properties of the heat shrinkable tape were measured considering the high likelihood of electrochemical corrosion in the complex ocean environment (Table 2 and Figure 8). The results show that the heat shrinkable tape had good cathodic disbondment properties in various test condition and outdistanced the standard of commercial heat shrinkable tapes (cathodic disbondment ＜5 mm, ≤ 12 mm, and ≤ 5 mm on the condition of 23°C×28 days, 110°C×28 days, and 65°C×48 h, respectively), probably benefitting from the barrier protection of the crystallization region.

Table 2. The properties of the crosslinked-polypropylene heat shrinkable tape

Property		Test condition	Result
Peel strength (N/mm)	With the steel painted the epoxy primer	23°C	12.3
		110°C	4.4
	With the polypropylene anticorrosive coating	23°C	12.1
		110°C	4.4
Impact strength (J/mm)		23°C	＞10
Cathodic disbondment (mm)		23°C×28 days	3.2
		110°C×28 days	4.5
		65°C×48 h	2.2

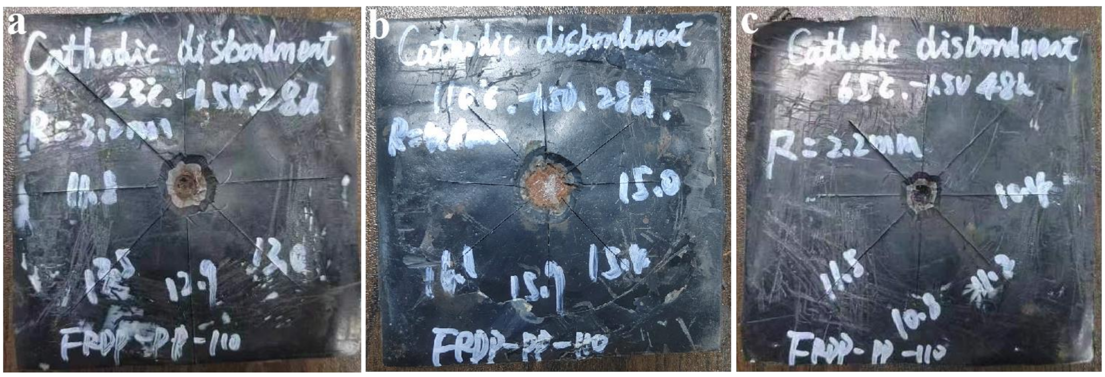

Figure 8. Cathodic disbondment of the crosslinked-polypropylene heat shrinkable tape: (a) 23°C×28 days, (b) 110°C×28 days, (c) 65°C×48 h

4. Conclusions

In summary, using PPB, mPPR, OBC, and mPE as matrix, and the radiation-crosslinked polypropylene was prepared by one-step process and used to produce the heat shrinkable tape further. The results show that the crosslinked-polypropylene has optimum overall performance when the amounts of PPB, mPPR, OBC, and mPE are 50%, 10%, 15%, and 15%, respectively. The SEM result proves that the one-step process can effectively carry out the orientation of the polypropylene and is conducive to the enhancement of its crystallization and the improvement of the performances. With the increase of the irradiation dose, the crosslinking density of the polypropylene increases but its degree of crystallinity reduces. The appropriate irradiation dose is 6 Mrad, benefitting to the balance between the crosslinking density and the degree of crystallinity, and the tensile strength, the elongation at break, the shrinkage ration, the gel content of the crosslinked-polypropylene sheets reach 33 MPa, 390%, 30%, 51%, respectively. The tensile strength of the samples after heat treatment enhanced slightly and the elongation at break improved observably, benefitting to minishing the weld seam sets and enhancing the corrosion prevention. The crosslinked-polypropylene had excellent high temperature resistance, and the decrease of its tensile strength and elongation at break did not exceed 15% after thermal ageing at 130°C for 100 days. Moreover, the corresponding crosslinked-polypropylene heat shrinkable tapes at both room temperature and high temperature show outstanding peel strength and impact strength, and its cathodic disbondment properties are also good in various test condition.

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