Construction and application research of FTIR analysis method for internal epoxy resin coated plastic lined steel pipe materials

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Abstract: The service performance of internally coated epoxy resin composite steel pipes critically depends on the material and quality of their plastic coating layer. In response to the challenges posed by infrared spectroscopy identification difficulties arising from filler interference, poor surface conditions, or component migration in practical testing, this paper systematically compares the applicability of Attenuated Total Reflection (ATR) and potassium bromide (KBr) pellet-pressing methods in the analysis of plastic coating layers. The results indicate that the ATR method is simple to operate and suitable for rapid initial screening of samples with clean surfaces and minimal filler interference. Conversely, the KBr pellet pressing method effectively eliminates surface selectivity effect, clearly presenting the characteristic absorption peaks of the bulk resin and additives (such as phthalate plasticizers, calcium carbonate, barium sulfate, etc.), making it particularly suitable for arbitration analysis of complex formulations or poor-quality ATR spectra. Based on this, this paper establishes a standardized infrared identification process of "ATR initial screening - KBr verification", and verifies the reliability of this process in identifying bisphenol A epoxy resin, polyester-epoxy hybrid systems, and other aspects through typical samples. The research findings provide practical and efficient analytical technical support for quality control, failure analysis, and market supervision of internally coated composite steel pipes.

Key words: internally coated epoxy resin composite steel pipe; plastic coating layer; Fourier Transform infrared spectroscopy (FTIR); Attenuated Total Reflection (ATR) method; KBr pellet method; material identification; bisphenol A epoxy resin; polyester-epoxy hybrid system

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Epoxy resin coated steel pipe is a new type of pipe material formed by coating epoxy resin powder on the inner wall of steel pipe through a special process and curing at high temperature. This structure combines the excellent mechanical properties of metal steel pipes with the superior corrosion resistance of polymer epoxy resin, which not only effectively resists chemical medium erosion and inhibits microbial adhesion, but also significantly reduces fluid conveying resistance due to its smooth inner wall. Based on these characteristics, this type of pipe material is widely used in applications where high requirements for corrosion prevention, scale prevention, and medium purity are needed, such as building water supply, fire protection systems, municipal pipe networks, petrochemical industry, and electric power energy.

The material selection, curing state, and uniformity of the plastic coating layer have a decisive impact on the service performance of composite steel pipes. Currently, there is a wide variety of commercially available internal epoxy resin

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Biography: Yu Qiaoling (1986-), holds a master's degree and is a senior engineer, primarily engaged in the inspection, testing, and development of testing methods for polymer materials and products.

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coatings, including bisphenol A-type epoxy resin, phenolic epoxy resin, and various modified systems (such as toughened and high−temperature resistant types). Among them, bisphenol A−type epoxy resin is the most predominant (see Figure 1 for its structural formula). Coatings prepared with different formulations and curing processes exhibit significant differences in chemical resistance, temperature resistance, and mechanical properties. However, issues such as the use of inferior resins and improper control of curing processes often occur in practical engineering, leading to coating peeling, blistering, or early failure of pipelines during operation, which seriously affects engineering safety. Therefore, developing an efficient and accurate method for identifying and evaluating the quality of plastic coating materials is of great significance for ensuring the quality of pipe materials, conducting failure analysis, and regulating market order.

Traditional coating analysis methods, such as chemical analysis, are cumbersome and destructive; thermal analysis techniques (such as DSC, TGA) can reflect the thermal behavior of materials, but have limited ability to identify chemical structures; scanning electron microscopy (SEM) can observe surface morphology, but cannot obtain chemical composition information. In contrast, Fourier transform infrared spectroscopy (FTIR) technology can provide rich molecular structure information (such as functional group types and chemical bond structures), with advantages such as fast analysis speed, small sample requirements, and convenient operation. It has become one of the core methods for qualitative analysis of polymer materials. However, applying FTIR technology to the quality control of internally coated epoxy resin composite steel pipes still faces challenges: firstly, the coating is tightly bonded to the steel substrate, making sampling and sample preparation difficult; secondly, the presence of fillers and other additives interferes with the accurate identification of characteristic functional groups.

For this purpose, this study focuses on the plastic layer characteristics of internally coated epoxy resin composite steel pipes, systematically investigates key aspects such as sampling strategies, sample preparation processes, and optimization of testing parameters, establishes the correspondence between characteristic infrared absorption peaks and resin types, and verifies the reliability of the testing method through testing actual market samples. Ultimately, a standardized operating procedure is formed, and a complete technical system from rapid screening to precise arbitration is constructed, providing practical basis for the scientific identification of plastic coating materials.

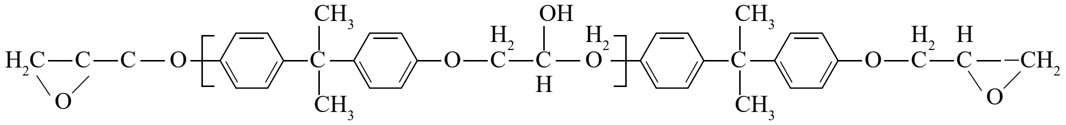


Figure 1 Structural formula of bisphenol A epoxy resin (E-type epoxy resin)

1 Experimental part

1.1 Materials and instruments

Epoxy resin-coated composite steel pipe: commercially available; Fourier transform infrared spectrometer: NICOLET 6700, Thermo Fisher, USA; Spectroscopically pure potassium bromide: Aladdin; Hydraulic press: Shanghai Shanyue Scientific Instrument Co., Ltd.

1.2 Experimental methods

1.2.1 Sample preparation

Cut a test piece of approximately 10 cm × 10 cm from each composite steel pipe. Wipe the surface of the plastic coating layer with anhydrous ethanol and acetone in order to remove any possible oil stains and dust, and let it dry at room temperature for future use.

1.2.2 Attenuated total reflection (ATR) method

Scrape a plastic-coated layer sample from the composite steel pipe, place the sample on the Ge crystal attached to the ATR, and apply a fixed pressure for testing. Test parameters: scanning range 4000~600 cm^{-1}, resolution 4 cm^{-1}, scanning 32 times. Before testing each sample, clean the crystal background with anhydrous ethanol and collect the background spectrum. All spectral data undergo automatic baseline correction and atmospheric background subtraction.

1.2.3 Potassium bromide (KBr) pellet pressing method

Using a file, polish the plastic-coated layer of the composite steel pipe into fine powder. Take 1~2 mg of powder sample, mix it thoroughly with about 200 mg of dried KBr powder in an agate mortar, and grind until it is fine and uniform. Press it into a transparent or translucent thin sheet under a pressure of 25 MPa, and then proceed with scanning tests. Test parameters: scanning range 4000~400 cm^{-1}, resolution 4 cm^{-1}, scanning 32 times. Complete the test by calibrating the baseline and identifying characteristic peaks.

2 Results and discussion

2.1 ATR method

Figure 2 displays the infrared spectrum of the plastic-coated layer on the composite steel pipe, measured using the ATR method. Through systematic analysis of the spectrum, the main chemical composition and structural characteristics of the plastic-coated layer can be identified.

In the characteristic frequency region (4,000 cm^{-1}~ 1,300 cm^{-1}), the spectrum displays the typical absorption peaks of bisphenol A epoxy resin. The absorption peaks located at 2,964 cm^{-1} and 2,930 cm^{-1} are attributed to the C—H stretching vibrations of methyl (—CH_3) and methylene (—CH_2—) in the aliphatic chain, respectively, providing strong evidence for the alkyl structure in the polymer backbone. In the aromatic ring vibration region, a series of absorption peaks at 1,607 cm^{-1}, 1,580 cm^{-1}, and 1,508 cm^{-1} originate from the C═C backbone vibrations of the benzene ring, clearly indicating the presence of an aromatic ring structure. Notably, the double peaks at 1,384 cm^{-1} and 1,363 cm^{-1} are characteristic of the symmetric bending vibrations of geminal dimethyl (—$C(CH_3)_2$—), which is a signature unit of the bisphenol A monomer structure. In the ether bond vibration region, the absorption peak at 1,296 cm^{-1} is identified as the asymmetric stretching vibration of aromatic ether (Ar—O—C), while the strong peak at 1,234 cm^{-1} corresponds to the asymmetric stretching vibration of C—O—C in aromatic ether (Ar—O—Ar). Overall, the broad absorption band from 1,300 to 1,000 cm^{-1} is mainly attributed to the stretching vibration modes of various C—O bonds in the molecule. Furthermore, in the out–of–plane bending vibration region of the benzene ring C—H, the absorption peaks at 828 cm^{-1} and 802 cm^{-1} further confirm the para-disubstituted mode of the benzene ring. All these characteristic peaks mutually corroborate and collectively constitute the standard infrared fingerprint spectrum of bisphenol A epoxy resin, confirming that the base resin of the plastic coating layer is of this type.

It is worth pointing out that in the ATR spectrum, the overall absorption intensity in the 4000~1300 cm^{-1} region is generally weak. This phenomenon is not due to insufficient sample concentration, but is closely related to the physical mechanism of ATR technology itself. The intensity of the ATR signal depends on the penetration depth of the evanescent field generated by infrared light in the sample, which is jointly influenced by the wavelength of the incident light, the refractive index of the ATR crystal, and the refractive index of the sample. In the high wavenumber region (i.e., short wavelength region), the penetration depth significantly decreases, resulting in a weakened signal response. Therefore, in ATR mode, one cannot directly infer the absolute content of functional groups based solely on peak intensity. A comprehensive judgment combining standard spectra and structural information is required.

In addition to the main body of epoxy resin, the spectrum also displays characteristic signals of other additives in the fingerprint region (<1,300 cm^{-1}). The absorption peaks located at 987 cm^{-1}, 636 cm^{-1}, and 610 cm^{-1} are characteristic of the stretching and bending vibrations of the S—O bond in sulfate (SO_4^{2-}), indicating the possible presence of a small amount of inorganic sulfate fillers or impurities in the sample. More importantly, a set of absorption peaks at 1,041 cm^{-1}, 779 cm^{-1}, 739 cm^{-1}, and 696 cm^{-1} strongly suggests the addition of phthalate plasticizers to the sample. Among them, the peaks at 779 cm^{-1} and 739 cm^{-1} are particularly crucial, as they originate from the out–of–plane bending vibrations of the C—H bond in the ortho-disubstituted benzene ring of phthalate molecules, which are the most distinct fingerprint characteristics of this type of plasticizer. Typically, phthalates exhibit a stretching vibration peak of the ester carbonyl (C═O) bond near approximately 1,740 cm^{-1}. However, in this ATR spectrum, this expected peak position is not clearly visible. This absence may be attributed to multiple factors: on the one hand, the response of ATR technology to strongly polar groups (such as C═O) may be limited by surface contact and refractive index matching;

on the other hand, if the amount of plasticizer added is low, its C═O absorption peak may be obscured by the broad absorption band of the epoxy resin backbone, or it may be difficult to distinguish due to peak overlap.

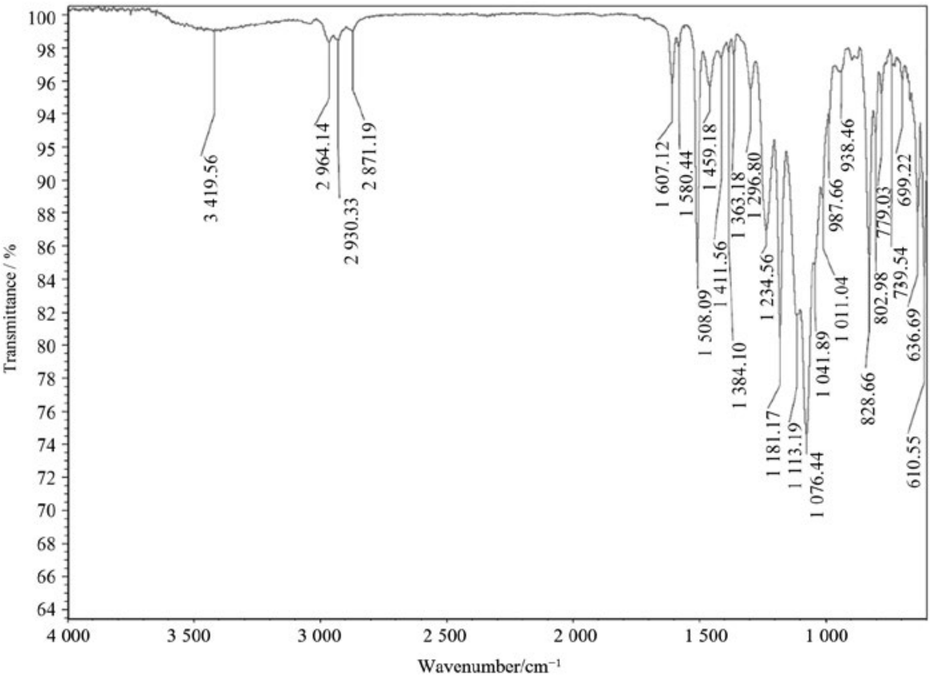


Figure 2 ATR infrared spectrum

2.2 KBr pellet pressing method

Figure 3 displays the infrared spectrum of the plastic-coated layer on the composite steel pipe, measured using the KBr pellet method. Compared to the Attenuated Total Reflection (ATR) method, the KBr pellet method involves uniformly dispersing the sample in a potassium bromide matrix and compressing it into a transparent pellet for transmission measurement. This fundamentally avoids the signal attenuation and surface selectivity enhancement effects caused by the limited penetration depth of evanescent waves in ATR technology. This classical method exhibits significant advantages in terms of band intensity, peak shape resolution, and feature combination, especially in the characteristic frequency region of 4000~1300 cm^{-1}. It can more accurately and comprehensively reflect the bulk chemical composition and molecular structure information of the plastic–coated layer material, effectively avoiding spectral deviations and misinterpretations caused by physical factors such as surface component enrichment, crystallinity gradients, or molecular orientation.

Through systematic analysis of the spectrum, the characteristic absorption peaks of each chemical component in the coating layer can be clearly identified. The broad and gentle absorption band at 3423 cm^{-1} is attributed to the stretching and vibrating of hydroxyl groups (O—H), which may originate from residual hydroxyl groups in the resin, by–products of the curing reaction, or adsorbed environmental moisture. The weak absorption at 3035 cm^{-1} corresponds to the stretching and vibrating of =C—H bonds on the aromatic ring, providing direct evidence of the presence of benzene ring structures. The absorption peaks at 2 964 cm^{-1} and 2 929 cm^{-1} are attributed to the C—H stretching and vibrating of methyl (—CH_3) and methylene (—CH_2—) groups in the aliphatic chain, reflecting the alkyl structure in the resin skeleton and additives. The characteristic double peaks at 1384 cm^{-1} and 1362 cm^{-1} are due to the symmetric bending vibrations of geminal dimethyl (—$C(CH_3)_2$—), which are typical markers of bisphenol A structural units. The C=C backbone vibrations of the aromatic ring exhibit a series of clearly distinguishable absorption peaks at 1607 cm^{-1}, 1581 cm^{-1}, and 1509 cm^{-1}. The absorption peaks at 1297 cm^{-1} and 1245 cm^{-1} are attributed to the asymmetric stretching and vibrating of C—O—C bonds in aromatic ethers (Ar–O–C) and diaryl ethers (Ar–O–Ar), respectively, constituting key vibrational modes of the epoxy resin network skeleton. The broad and strong absorption band between 1300 and 1000 cm^{-1} is mainly formed by the superimposed contributions of the stretching and vibrating of various C—O bonds (including ether bonds, ester bonds, etc.) in the system. In addition, the absorption peaks at 827 cm^{-1} and 800 cm^{-1} further confirm the out–of–plane bending vibration mode of C—H in the para-disubstituted benzene ring. The collaborative identification of the above series of characteristic peaks collectively constitutes an unambiguous infrared fingerprint spectrum of bisphenol A epoxy resin, confirming its role as the basic film-forming substance in the coating layer.

In addition to the main resin, the spectrum clearly reveals the presence of various functional additives. The strong absorption peak at 1737 cm^{-1} is attributed to the stretching vibration of ester carbonyl (C═O). Combined with the stretching vibration of C—O in the 1122 cm^{-1} and 1040 cm^{-1} regions, as well as the out–of–plane bending vibration combination of ortho-disubstituted benzene rings at 778 cm^{-1}, 727 cm^{-1}, and 695 cm^{-1}, it can be unambiguously identified as the addition of phthalate plasticizers. On the other hand, the sharp absorption peak at 983 cm^{-1} corresponds to the symmetric stretching vibration of sulfate ions (SO_4^{2-}), with asymmetric bending vibrations at 637 cm^{-1} and 610 cm^{-1}. The low-frequency absorptions at 559 cm^{-1}, 513 cm^{-1}, and 459 cm^{-1} originate from the mode splitting of symmetric bending vibrations due to crystal field effects. This complete infrared characteristic sequence of sulfate salts confirms the use of barium sulfate ($BaSO_4$) as a functional filler. Crucially, the sharp absorption peak at 1888 cm^{-1} is the most distinctive high-frequency characteristic of C═O stretching vibration in anhydride curing agents, providing decisive evidence for the adoption of the anhydride curing pathway in this epoxy system.

The introduction of the aforementioned three components serves specific material function design and performance optimization goals: phthalate plasticizers, by intercalating into the polymer chains, weaken intermolecular forces and enhance segmental mobility, thereby effectively improving the inherent hard and brittle properties of epoxy resin, significantly enhancing the coating's flexibility, impact resistance, and adhesion to steel substrates; barium sulfate fillers, by constructing an organic–inorganic composite system, synergistically leverage the toughness of polymers and the rigidity of inorganic fillers, improving the coating's mechanical strength, hardness, wear resistance, and impermeability, and optimizing the rheological behavior and application performance of the coating; while anhydride curing agents, through their ring–opening addition reaction with epoxy groups, form a highly cross–linked three-dimensional network structure dominated by ester bonds, endowing the coating with excellent heat resistance, chemical stability, electrical insulation, and long–term durability.

2.3 Test process construction and case analysis

In routine testing, if only the main material of internally coated epoxy resin composite steel pipes needs preliminary identification, it is recommended to prioritize the use of the ATR method, which is easy to operate. When the matching degree between the obtained spectrum and the standard infrared spectral library is less than 80%, manual comparison with typical characteristic peaks can be conducted to assist in the determination. If the quality of the ATR spectrum is unsatisfactory due to poor surface conditions of the sample or interference from inorganic fillers, the KBr pellet method should be used for retesting to obtain more accurate information about the bulk material. The following examples involving two different samples are provided for illustration.

Firstly, the infrared spectroscopy analysis of the sample coating layer was conducted using the ATR method, and the results are shown in Figure 4. The infrared characteristic peaks of Sample 1 are clearly attributed: 2970 cm^{-1} and 2925 cm^{-1} correspond to the stretching and bending vibrations of $—CH_3$ and $—CH_2—$; 1606 cm^{-1}, 1580 cm^{-1}, and 1508 cm^{-1} correspond to the stretching vibrations of the aromatic ring skeleton (C═C); 1295 cm^{-1}, 1235 cm^{-1}, 1181 cm^{-1}, 1104 cm^{-1}, 1085 cm^{-1}, and 1037 cm^{-1} correspond to the stretching and bending vibrations of C—O—C and C—O; 828 cm^{-1} reflects the bending vibration of C-H in the para-substituted benzene ring, all of which are typical absorptions of epoxy resin. In addition, the broad and gentle absorption peaks at 875 cm^{-1} and 1416 cm^{-1} are attributed to the filler calcium carbonate.

The ATR spectrum of Sample 2 was strongly interfered by inorganic fillers, obscuring the characteristic peaks in the 1,500~1,300 cm^{-1} region. Additionally, the low intensity of aromatic ring skeleton vibrations (1,600 cm^{-1}, 1,500 cm^{-1})

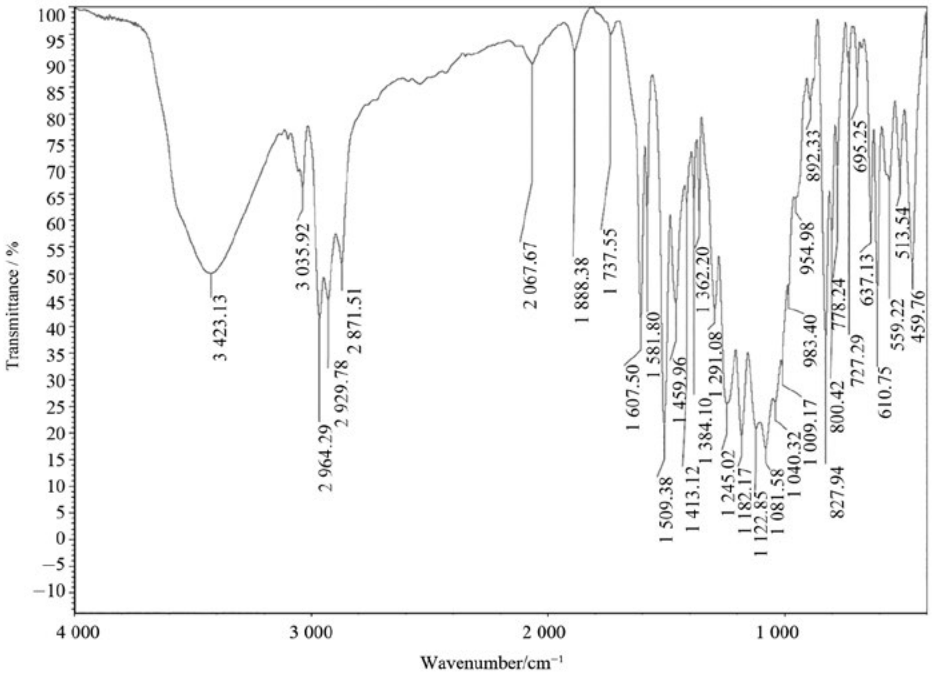


Figure 3 Infrared spectrum of KBr pellet method

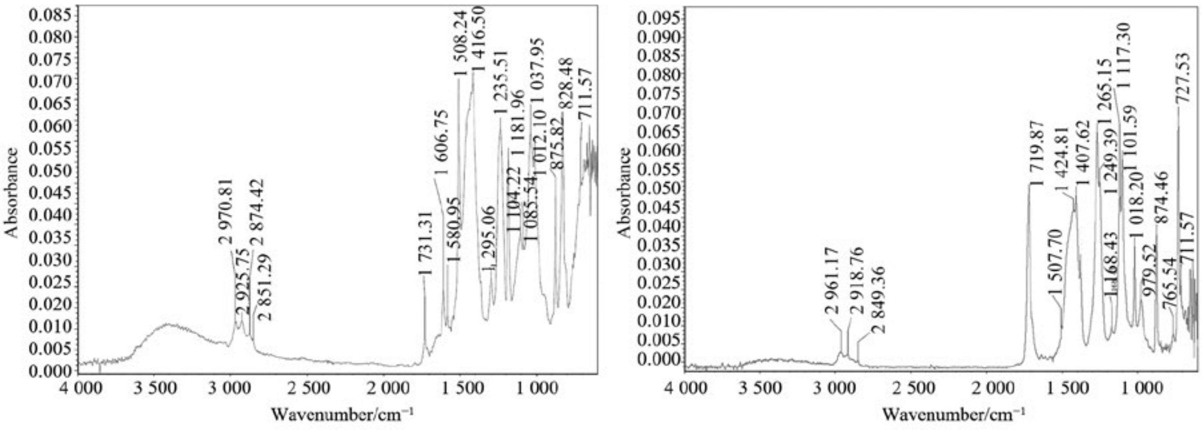
resulted in a high degree of similarity to polyester (PET) in the preliminary spectrum. After retesting using the KBr pellet method, key features became clearly visible: 2,965 cm^{-1}, 2,875 cm^{-1}, 1,610 cm^{-1}, 1,577 cm^{-1}, 1,505 cm^{-1}, 1,456 cm^{-1}, 1,246 cm^{-1}, 1,171 cm^{-1}, 1,077 cm^{-1}, and 829 cm^{-1} were attributed to epoxy resin absorption; 1,720 cm^{-1} (ester group C=O stretching vibration), 1,267 cm^{-1}, 1,118 cm^{-1}, 1,101 cm^{-1}, 1,017 cm^{-1}, 978 cm^{-1}, and 727 cm^{-1} were attributed to polyester absorption; broad and strong absorptions at 2,513 cm^{-1}, 1,791 cm^{-1}, 875 cm^{-1}, 712 cm^{-1}, and 1,429 cm^{-1} were attributed to calcium carbonate; and 632 cm^{-1} and 609 cm^{-1} indicated the presence of sulfate in the sample.

It is worth noting that some coating layers utilize a "polyester-epoxy resin" composite system. In such systems, infrared spectroscopy often reveals a phenomenon where the characteristic peak intensity of polyester is higher than that of epoxy resin. There are two primary reasons for this: firstly, the proportion of polyester resin added in the formulation is relatively high; secondly, during the curing process, due to differences in surface tension and compatibility between the two phases, the polyester component tends to migrate more easily to the surface layer of the coating, thus selectively enhancing the signal in FTIR detection. The design of this composite system aims to achieve synergistic optimization of performance: on the one hand, it can significantly enhance the flexibility, impact resistance, and adhesion to steel substrates of the coating; on the other hand, it helps to reduce raw material costs. However, compared to pure epoxy systems, it exhibits weaker chemical resistance (especially alkali resistance) and long-term thermal stability, while posing higher requirements on formulation design, component compatibility control, and production process parameters.

3 Conclusion

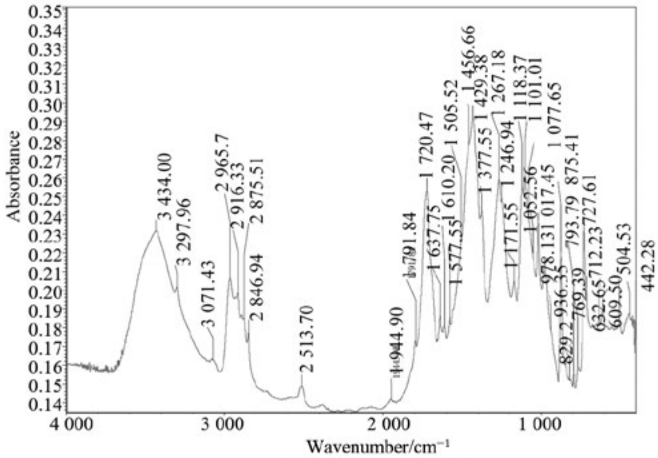
(1) The Attenuated Total Reflection (ATR) method is suitable for the rapid preliminary identification of the plastic coating layer on internally coated epoxy resin composite steel pipes. It has advantages such as no need for sample preparation and convenient operation. However, its signal is easily affected by surface conditions, filler interference, and component migration. Especially in the high wavenumber region, its sensitivity is relatively low, which may lead to missed detection or misjudgment of key functional groups.

(2) The KBr pellet method, which obtains bulk information through transmission mode, can more authentically and comprehensively reflect the infrared characteristics of the epoxy resin matrix (such as bisphenol A structural units, aromatic ether bonds, para-substituted benzene rings, etc.),



a-Sample 1: ATR method

b-Sample 2: ATR method



c-Sample 2: KBr pellet method

Figure 4 Infrared spectroscopy analysis of plastic coating layer

functional additives (such as phthalate plasticizers, calcium carbonate, barium sulfate), and curing agent types (such as anhydrides) in the plasticized coating layer. It is a reliable arbitration method for complex systems or controversial samples.

(3) For the common "polyester–epoxy resin" mixed system in actual samples, the ATR method tends to overestimate the content of the polyester component due to its surface enrichment, and a comprehensive determination is required in conjunction with the KBr pellet method.

(4) The two–tier identification process of "ATR initial screening + KBr re–examination" proposed in this paper balances efficiency and accuracy, effectively addressing challenges such as material mixing and complex formulations in commercially available products. It provides a standardized and scalable infrared spectroscopy analysis technique for quality supervision and failure traceability of internally coated composite steel pipes.