ISSN 2616-5880 Vol. 6, Issue 2: 37-42, DOI: 10.25236/AJMC.2025.060205

Determination and Sensitivity Analysis of Total Ethylene Content Based on Fourier Infrared Method

Chunmiao Jiang

Sino-Korea (Wuhan) Petrochemical Co., LTD, Wuhan, China
jiangcm.zhsh@sinopec.com

Abstract: *Total ethylene content (TEC) is an important indicator for evaluating the impact strength of impact polypropylene copolymer products. It provides guidance for controlling parameters such as feed ratio, copolymerization time, and feed rate in processing. Currently, the measurement of ethylene content by Fourier transform infrared (FTIR) spectrometry is the most widely used method in the industry because of its high speed, excellent repeatability, and the wealth of information it provides. In this paper, a method based on FTIR spectroscopy and a standard curve for determining the TEC is proposed, and the sensitivity of the method under various conditions is analyzed. Precision evaluation on standard samples with known TEC indicates that the proposed method achieves a bias of –0.09 and an RMSE of 0.26. Furthermore, sensitivity analysis shows that when the sample thickness error is within 5μm, the error in TEC determination is less than 0.3%, and when the peak area error in the 707–750 cm^{-1} region is within 0.006, the error is less than 0.006%. This method is currently applied at the Sino–Korean Petrochemical Analysis Center and plays an important role in providing accurate analytical data, guiding process production, and enhancing product market competitiveness.*

Keywords: *FTIR spectroscopy, impact polypropylene copolymer, total ethylene content, sensitivity analysis*

1. Introduction

Total ethylene content (TEC) is a key parameter in assessing the impact resistance and overall quality of impact polypropylene copolymers, as it directly influences processing parameters such as feed ratio, copolymerization time, and feed rate [1]. In recent years, Fourier transform infrared (FTIR) spectroscopy has emerged as a preferred analytical technique due to its rapid analysis speed, high repeatability, and minimal sample requirements [2,3].

Several studies have specifically explored the application of FTIR in ETC measurement within polypropylene copolymers. Zhang et al. investigated the influence of heating temperature, pressure, and compression time on the measured ethylene content in polypropylene copolymers while ensuring bubble-free film formation [4]. Their study determined the optimal conditions for film preparation to achieve accurate ethylene quantification. Similarly, Sun et al. analyzed the effects of different cooling methods, antioxidants, and particle size on the ethylene content measurement in ethylene-propylene copolymer polypropylene powders [5]. Their research provided insights into optimizing hot-press sample preparation for improved accuracy in ethylene detection. However, challenges remain in mitigating measurement uncertainties—particularly those related to variations in sample thickness and peak area—that can affect the accuracy of ethylene content determination.

In this study, we propose a FTIR-based method , establishing a standard curve from eight groups of standard samples and 24 test datasets. Validation of the proposed method is performed based on 12 sets of experimental data. In addition, a comprehensive sensitivity is employed to assess the robustness of the algorithm. This work not only provides a rapid and accurate approach for determining TEC but also offers valuable guidance for optimizing production processes and reducing the risk of product nonconformity due to analytical errors.

ISSN 2616-5880 Vol. 6, Issue 2: 37-42, DOI: 10.25236/AJMC.2025.060205

2. Measurement method

2.1 Method principle

The measurement principle is based on infrared absorption spectroscopy. When a sample is irradiated with infrared light of continuously varying frequency, its molecules absorb radiation at specific frequencies corresponding to vibrational or rotational transitions. These transitions cause transient changes in the dipole moment, leading to excitation from the ground state to higher energy states. As a result, the transmitted light intensity decreases at these frequencies, forming a spectrum that plots transmittance (T%) against wavenumber or wavelength—this is the infrared spectrum. Using this principle, the molecular composition and structure of a sample can be analyzed. In ethylene content measurement, ethylene molecules exhibit characteristic infrared absorption peaks, with absorption intensity correlating to concentration. By measuring the absorption within specific wavenumber ranges, ETC can be quantitatively analyzed [3].

2.2 Main instruments

1) FTIR Spectrometer: Bruker model INVENIO S

2) Software: OPUS

3) Measurement Range: 400–6000 cm^{-1}

2.3 Operating procudure

1) Preparation of Standard Sample

(a) Cut three pieces of aluminium foil with a thickness of 0.1 mm, mark cross-shaped scratches on them, and clean them thoroughly with anhydrous ethanol.

(b) Select one piece of aluminium foil, fold both its left and right sides inward twice—leaving a central area with a width of 35–40 mm—and weigh out approximately 2g of pre-melted sample strip to place in the central area.

(c) Cover the aluminium foil containing the sample with the remaining two pieces of aluminium foil, place the assembly between two iron plates, and load it into the press machine.

(d) Choose the "PPGB 210" method, confirm that the heating temperature is 210 ± 10℃, and set the working pressures to 10 and 300 mPa with corresponding operating times of 5 minutes each; then press the start button to begin.

(e) As soon as the pressing is complete, immediately remove the aluminium foil assembly and immerse it in cold water for about 10 seconds.

(f) Finally, take out the assembly and carefully peel off the sample film.

2) Construction of the Standard Curve

For each sample, three peak area values in the 707–750 cm^{-1} wavenumber range are obtained. This yields a linear relationship as shown in Table 1 and Fig. 1.

Table 1 Total ethylene content and peak area/thickness value of the standard sample

Standard Sample	TEC /%	Peak area/thickness
BC03CBQ	4.28	0.011
BC02NQ	9.46	0.024
BC6DQ	6.38	0.016
BC6DRQ	8.96	0.022
PN2100X	11.18	0.029
PN4100	16.90	0.040
PN6120X	11.56	0.028
BC03HRQ	8.78	0.023

ISSN 2616-5880 Vol. 6, Issue 2: 37-42, DOI: 10.25236/AJMC.2025.060205

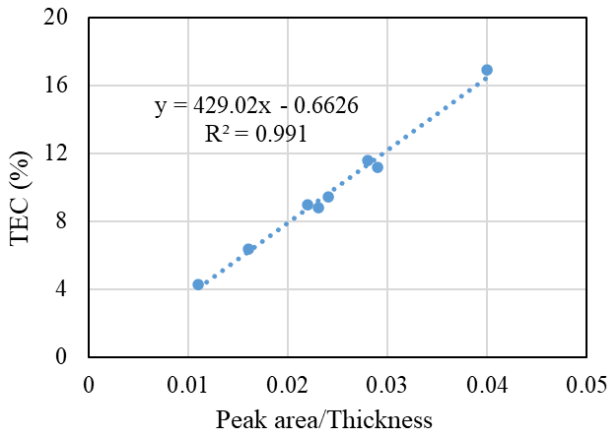

Fig.1 Linear curve of total Eehylene content vs. peak area/thickness

From Fig. 1, the standard curve can be expressed as:

$$Q=430*S/T-0.7759 \quad (1)$$

where Q is the TEC, S is the measured peak area, and T is the sample thickness. The coefficient of determination (R^2) is close to 1, indicating a strong linear relationship. This equation is effective for accurately determining the TEC of samples at standard concentrations and is suitable for data extraction.

3. Precision Verification of the Standard Curve

ETCs were calculated from these data using the proposed formula (1), and the average difference between the measured values and the standard values was calculated to determine the precision of the curve, as shown in Table 2.

Table 2 Comparison Between Standard and Measured Total Ethylene Content (%)

Sample	Thickness	Peak Area	Standard TEC	Measured TEC	Difference Between Standard and Measured Value
1	250	2.6470	4.28	3.80	(0.48)
	248	2.7850	4.28	4.07	(0.21)
2	247	5.8710	9.46	9.47	0.01
	252	6.1210	9.46	9.69	0.23
3	252	4.2320	6.38	6.47	0.09
	250	4.2330	6.38	6.53	0.15
4	252	5.7800	8.96	9.11	0.15
	250	5.5659	8.96	8.82	(0.14)
5	252	10.1290	16.90	16.53	(0.37)
	250	9.9710	16.90	16.40	(0.50)
6	251	7.1590	11.56	11.51	(0.05)
	250	7.1710	11.56	11.58	0.02
				Bias	(0.09)
				RMSE	0.257235709

ISSN 2616-5880 Vol. 6, Issue 2: 37-42, DOI: 10.25236/AJMC.2025.060205

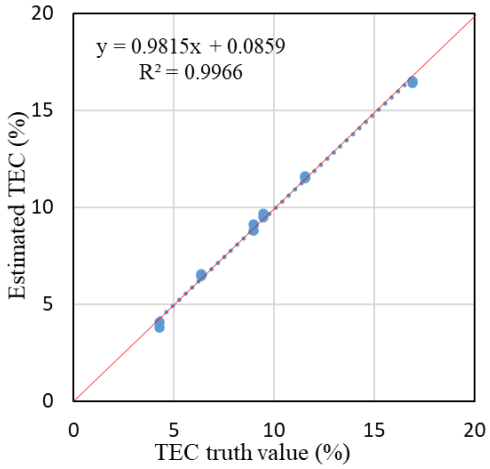

Fig.2 Comparison Between Standard and Measured Total ethylene content (%)

As shown in Fig.2, there are good consistence between the estimated TEC and the truth value. The results indicate that the average difference (bias) is –0.09% with an RMSE of approximately 0.26, which demonstrates high precision and good repeatability of the proposed method.

4. Sensitivity anlysis

Since the estimation of ethylene content relies on sample thickness and peak area as input parameters, any errors arising from nonuniform pressing, instrument precision, or operator handling during thickness measurement—and the inherent uncertainty in FTIR peak area measurement—may affect the result.

A simulation was conducted under various conditions by setting:

- Four TEC levels from 4% to 16% at 4% intervals.
- Twenty-one thickness values from 200μm to 300μm at 5μm intervals.
- Twenty-one peak area values from 2 to 12 at 0.5 intervals.

For the measurement errors:

- Twenty-one thickness error values were defined from –5μm to 5μm at 0.5μm intervals.
- Twenty-five peak area error values were defined from –0.006 to 0.006 at 0.0005 intervals.

The sensitivity analysis is based on the following general formulation:

$$\triangle Q_s = Q(S + \triangle S, T) - Q(S, T) \quad (2)$$

$$\triangle Q_t = Q(S, T + \triangle T) - Q(S, T) \quad (3)$$

where S and T represent the peak area and sample thickness, respectively; $\triangle S$ and $\triangle T$ are their corresponding measurement errors; and $\triangle Q_s$ and $\triangle Q_t$ denote the resulting errors in TEC due to these errors.

4.1 Sensitivity to Sample Thickness Error

(1)Observation 1: As the thickness error increases, the error in TEC also increases. For a 4% concentration sample, a thickness error of 1 μm leads to an error of −0.02%, 3 μm to −0.07%, and 4.5 μm to −0.10%. When the error exceeds 4.5 μm, the TEC error reaches approximately 0.12%.

(2)Observation 2: With constant sample thickness, higher concentration samples exhibit more pronounced increases in TEC error. For instance, at a thickness of 200 μm and a −5 μm error, the errors for 4%, 8%, 12%, and 16% samples are approximately 0.12%, 0.22%, 0.33%, and 0.43%, respectively.

(3)Observation 3: With a fixed thickness error, the TEC error slightly decreases as the sample thickness increases. For a 16% sample with a −3 μm error, the errors are 0.26%, 0.23%, 0.21%, 0.20%, 0.18%, and 0.17% for thicknesses of 200, 220, 240, 260, 280, and 300μm, respectively, as shown in Figure 3.

ISSN 2616-5880 Vol. 6, Issue 2: 37-42, DOI: 10.25236/AJMC.2025.060205

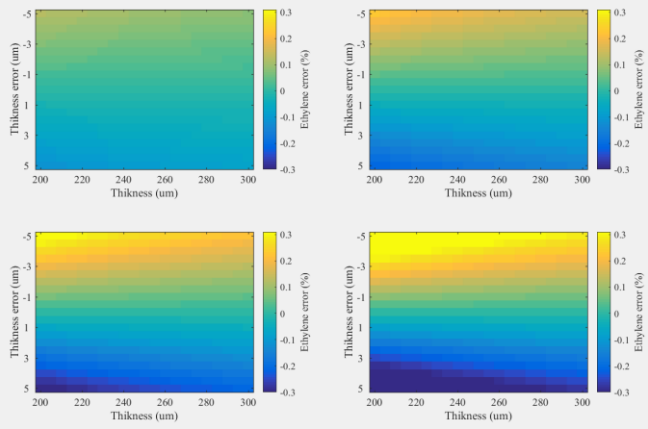

Fig.3 Sensitivity of total ethylene content estimation to sample thickness error

4.2 Sensitivity to Peak Area Error

(1)Observation 1: A smaller peak area error results in a smaller TEC error. As the peak area error approaches zero, the error in TEC also approaches zero; conversely, larger errors (indicated by a darker color in the simulation graphs) lead to increased TEC errors.

(2)Observation 2: For a constant peak area value, samples with higher TEC are more sensitive to peak area errors. For example, with a peak area of 4 and an error of 0.004, the errors for 4%, 8%, 12%, and 16% samples are approximately 0.005%, 0.009%, 0.013%, and 0.017%, respectively.

(3)Observation 3: With a fixed peak area error, larger peak area values yield lower TEC errors. For a 16% sample and a peak area error of -0.003, if the peak area values are 2, 4, 6, 8, 10, and 12, the corresponding errors are about 0.025%, 0.012%, 0.008%, 0.006%, 0.005%, and 0.004%, respectively, as shown in Figure 4.

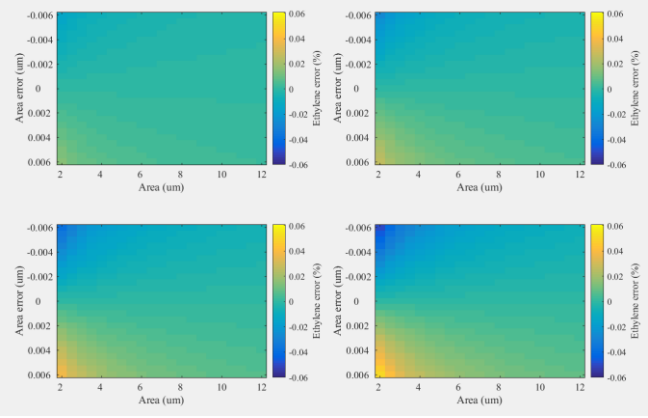

Fig.4 Sensitivity of total ethylene content estimation to peak area error

5. Conclusion

Based on eight groups of standard samples with different concentrations and 24 sets of test data, a standard curve was fitted to relate TEC with sample thickness and peak area in the 707–750 cm^{-1} wavenumber range. The resulting equation is Q=430*S/T-0.7759.

Using 12 sets of measured data from impact polypropylene copolymer samples, the accuracy of the formula was validated, yielding a bias of –0.09 and an RMSE of 0.26, which demonstrates the high precision of the proposed method. Based on this, the sensitivity of the TEC estimation model to the input parameters (peak area and sample thickness) was further analyzed. The sensitivity analysis showed that when the sample thickness error ranges from –5μm to 5μm, the model error is between –0.3% and 0.3%; and when the peak area error ranges from –0.006 to 0.006, the model error is between –0.06% and 0.06%.

Considering that in actual production our company typically requires an allowable error for TEC

ISSN 2616-5880 Vol. 6, Issue 2: 37-42, DOI: 10.25236/AJMC.2025.060205

ranging from ±0.1% to ±1% for different products, it can be concluded that the proposed method is capable of rapidly and accurately analyzing the TEC in impact polypropylene copolymer products. This provides effective guidance for adjusting process parameters, thereby reducing the risk of product nonconformity due to analytical data deviations.

Furthermore, based on simulation estimates, this paper also offers data references to help achieve an accuracy of less than ±0.1% with the current analytical model. Specifically:

- For samples with a 16% concentration, when the sample thickness error is less than 1.5μm with a thickness between 200 and 300μm, or when the thickness error is exactly 1.5μm with a thickness between 245 and 300μm, the TEC error is less than 0.1%.
- For samples with a 12% concentration, when the sample thickness error is less than 2μm with a thickness between 200 and 300μm, or when the thickness error is exactly 2μm with a thickness between 245 and 300μm, the TEC error is less than 0.1%.
- For samples with an 8% concentration, when the sample thickness error is less than 2.5μm with a thickness between 200 and 300μm, or when the thickness error is exactly 2.5μm with a thickness between 210 and 300μm, the TEC error is less than 0.1%.
- For samples with a 4% concentration, when the sample thickness error is less than 2.5μm with a thickness between 200 and 300μm, or when the thickness error is exactly 2.5μm with a thickness between 210 and 300μm, the TEC error is also less than 0.1%.

References

[1] Wang Huimin, Wang Haidong, Xu Lin, et al. Analysis of the factors affecting the determination of water content in ethylene oxide [J]. Brand & Standardization, 2025,(01):30-32.
[2] Wang Wei, Jiang Xiaohua. Introduction to Fourier Transform Spectrum Analysis Theory and Application [J]. Chemical Industry Times, 2013,27(09):38-41.
[3] Liang Yahui. Determination of Ethylene Content in Polypropylene Copolymer by Infrared Spectroscopy [J]. Chemical Enterprise Management, 2013,(12):90-91.
[4] Zhang Zhihua, Li Yongpeng, Zhang Zhenping, et al. Factors Affecting the Determination of Ethylene Content in Polypropylene Copolymer by Infrared Spectroscopy [J]. Henan Chemical Industry, 2023,40(11):54-55. DOI:10.14173/j.cnki.hnhg.2023.11.010.
[5] Sun Wei, Liu Yi, Liao Yunlong, et al. Study on influence factors for determination of ethylene content in polypropylene powder [J]. China Plastics, 2022,36(06):54-59. DOI:10.19491/j.issn.1001-9278.2022.06.009.