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Study on detection method for harmful chemical residues in regenerated cellulose food packaging materials

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Abstract: In order to accurately evaluate the migration risk of harmful elements in regenerated cellulose food packaging materials, a new method for detecting harmful chemical residues in regenerated cellulose food packaging materials was studied. By optimising the ratio of digestion reagents and procedures, complete digestion of the sample was achieved, and the optimal analytical spectral lines for Ti, Pb, Cr, and Cd were screened. The method validation results showed a good linear relationship ($r > 0.9989$) among the four elements, with a detection limit between $1.96\sim 12.94\ \mu\text{g}\cdot\text{kg}^{-1}$ and a quantification limit between $6.53\sim 43.19\ \mu\text{g}\cdot\text{kg}^{-1}$. The recovery rates ranged from 83.19% to 108.32%, with a relative standard deviation of 2.12–7.41%. The accuracy and repeatability were good. The results of testing five types of commercially available samples are all below the national standard limit. This method can provide technical support for the rapid screening and safety evaluation of harmful elements in regenerated cellulose packaging materials.

Keywords: regenerated cellulose; food packaging; harmful chemical residues; detection; safety evaluation; digestion pretreatment.

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1 Introduction

Regenerated cellulose, as a green and environmentally friendly food packaging material, is considered an ideal choice to replace traditional plastic packaging. During the processing, these materials typically require the use of various chemical reagents, including solvents, bleaching agents, plasticisers, etc., to improve their physical and chemical properties (Yin et al., 2025; Liu et al., 2025). If residual in the finished product, it will migrate upon contact with food, leading to allergic reactions and endocrine disruption. Therefore, developing a method with better detection performance for systematic screening and quantitative analysis of harmful chemical residues in regenerated cellulose food packaging materials is an important technical support for ensuring food safety and promoting the healthy development of the green packaging industry. Conducting relevant methodological research can help improve the quality control system of packaging materials and provide scientific basis for the formulation and revision of relevant regulations and standards (Zhang et al., 2025).

In recent years, many scholars have carried out research on the detection of harmful substances in packaging materials. Headspace gas chromatography technology has been widely used in this field because of its high efficiency and high sensitivity. For example, Subinor et al. (2025) established a headspace-gas chromatography for the simultaneous determination of 35 solvent residues in pharmaceutical packaging materials. The established method can effectively separate all the target compounds with high detection accuracy, which shows that headspace-gas chromatography has good applicability and reliability in the screening of multi-component solvents in complex matrices. This

technology is characterised by simple pretreatment and rapid analysis, and is suitable for batch screening of volatile organic solvents. Its shortcomings are that it is only suitable for volatile components, and it is impossible to detect the non-volatile or thermally unstable inorganic element residues that may exist in regenerated cellulose, and its anti-interference ability to complex substrates is limited. Yan et al. (2025) further established a method for the determination of 55 kinds of organic solvent residues in plastic drug packaging materials by headspace gas chromatography-mass spectrometry. By optimising the chromatographic column and headspace conditions, this method has achieved wider solvent coverage and more reliable qualitative and quantitative analysis under the selected ion monitoring and full scanning modes. The linear correlation coefficients of 55 solvents ranged from 0.9914 to 0.9999, the average recoveries ranged from 83.2% to 113.2%, and the relative standard deviations ranged from 1.61% to 7.72%. This method has high sensitivity and good accuracy, and is suitable for simultaneous detection of various trace organic solvents in complex matrix. However, its limitations are also concentrated in the category of volatile organic compounds, which cannot cover inorganic harmful residues such as heavy metals, and the analysis effect of strong polarity or high boiling point residues is not ideal. Wang et al. (2025) systematically studied the indirect detection technology of total fluorine content in food packaging materials. This paper systematically summarises the current methods of sample pretreatment and fluoride detection from three levels of detection principle, technical characteristics and application, and makes a comprehensive comparison and comprehensive evaluation of many technologies from the aspects of detection accuracy, precision, stability, time-consuming and economy. This work provides an important reference for understanding the technical path of fluorine-containing compounds detection, but it is mainly aimed at the total fluorine content, which cannot realise the qualitative and quantitative analysis of a single fluoride species, and the detection process is cumbersome, time-consuming, and requires high equipment and operation. Zhang et al. (2024) conducted in-depth research on the solvent residue and its migration behaviour in two typical plastic composite film bags for food. It is found that the nonconformity rate of solvent residues in the selected samples is as high as 90%. By simulating different storage conditions, it was observed that the total amount of solvent residues decreased significantly with the increase of time and temperature: after 18 months at room temperature, the total amount of solvent residues of the two samples decreased by 95% and 88.2% respectively; After being treated at 80°C for 1 h, the solvent in one sample was completely volatilised, and the total residue in the other sample was reduced by 95.8%. This study provides important data support for evaluating the safety of packaging materials in practical use, but its research object is traditional plastic composite film, and the material properties are significantly different from regenerated cellulose, so it is difficult to directly transfer the relevant conclusions.

To sum up, the existing detection methods have obvious shortcomings in the following aspects: first, the detection objects are limited, mostly concentrated on volatile organic solvents or total fluorine content, and there are few studies on synchronous screening of harmful inorganic elements such as titanium, lead, chromium and cadmium; Second, the matrix adaptability is poor. The existing pretreatment technology is mainly aimed at drug packaging or traditional plastics, and it is difficult to deal with the problems of incomplete digestion and element wrapping caused by complex matrices

such as porous, hydrophilic and silicate-containing fillers of regenerated cellulose. Third, the anti-interference ability is insufficient, and the optimisation strategy of spectral interference system for specific matrix of regenerated cellulose is lacking. Therefore, it is urgent to develop a comprehensive detection method which has strong adaptability, can cope with the influence of the unique matrix of regenerated cellulose, and has high sensitivity and multi-element synchronous detection ability.

Aiming at the above technical bottlenecks, this study systematically constructed a set of detection methods for harmful chemical residues in recycled cellulose food packaging materials based on microwave digestion and inductively coupled plasma atomic emission spectrometry (ICP-AES). The research objectives include: optimising pretreatment conditions to realise the complete digestion of regenerated cellulose matrix; Screening the best analytical spectral lines to suppress spectral interference; Sensitivity, accuracy and repeatability of the verification method; And applied to the screening of harmful elements in actual samples: the overall experimental scheme and feasibility demonstration are as follows:

- 1 *Introduction of ternary digestion system:* The traditional $\text{HNO}_3\text{-H}_2\text{O}_2$ system is difficult to completely decompose silicate fillers or coatings that may exist in the regenerated cellulose matrix, which easily leads to incomplete release of target elements due to being wrapped. In this study, $\text{HNO}_3\text{-HF-H}_2\text{O}_2$ ternary mixed digestion system was innovatively introduced. By setting different volume ratios (5 : 1 : 2, 5 : 2 : 1, 6 : 1 : 1) for comparison, the clarity and precipitation of digestion solution were used as evaluation indexes to select the optimal ratio. Based on the effective dissolving ability of hydrofluoric acid to silicate, this scheme can theoretically solve the problem of packing and ensure the complete release of target elements, which has a clear chemical basis.
- 2 *Optimisation of digestion procedure:* Under the condition of fixed microwave power, four temperature levels (90, 140, 180, 200°C) and three constant temperature time levels (10, 15, 20 min) were set, and their effects on digestion were systematically investigated. The gradient design covers the typical parameter range of conventional microwave digestion, which can give consideration to digestion efficiency and operational safety. The high temperature of 200°C can strengthen the oxidative decomposition of organic matrix, and the constant temperature time of 20 min can ensure the full reaction, avoid the long digestion cycle and ensure the feasibility and repeatability of the method in practical operation.
- 3 *Screening of spectral lines:* Aiming at the common problems of spectral overlap and matrix interference in simultaneous determination of multi-elements by ICP-AES, this study takes spectral line intensity, signal-to-background ratio and recovery rate as evaluation indexes, and screens the optimal analytical spectral lines for four elements, namely Ti, Pb, Cr and Cd, with the help of instrument background correction function. This strategy is different from the conventional method of relying solely on instrument database or literature recommended spectral lines, and can be personalised optimised according to the actual matrix characteristics of regenerated cellulose digestion solution, effectively reducing spectral interference and improving the selectivity and accuracy of detection.

- 4 *Verification and practical application of the method performance:* The method performance was systematically evaluated by linear range, detection limit, quantitative limit, recovery rate and precision, and applied to the actual detection of five kinds of commercial regenerated cellulose food packaging materials to verify its applicability in complex matrices. The verification scheme comprehensively covers the core indicators of methodology evaluation, and can comprehensively evaluate the reliability of the method from multiple dimensions such as sensitivity, accuracy and repeatability, so as to ensure the comparability and popularisation value of the research results.

To sum up, the technical route proposed in this study is fully reasonable and feasible in terms of chemical principle, experimental design and practical application, and it is expected to provide a reliable analytical tool for rapid screening and safety evaluation of harmful chemical residues in recycled cellulose food packaging materials.

2 Method for detecting harmful chemical residues in regenerated cellulose food packaging materials

This section systematically describes the material basis and operational specifications established by the method from the aspects of experimental materials and reagents, instrument equipment, sample pretreatment operation process, instrument working conditions, and standard solution preparation, providing a clear experimental framework for subsequent condition optimisation and method performance evaluation.

2.1 Materials and reagents for detecting harmful chemical residues

This experiment was conducted using commercially available regenerated cellulose food packaging materials. Blank polyethylene plastic samples were provided by Taiyuan Kaigong Plastic Company, and the information on other reagents used is shown in Table 1.

Table 1 Experimental reagent information table

<i>Reagent</i>	<i>Level</i>	<i>Purchase manufacturer</i>
Nitric acid	GR	Kermel Chemical Reagent Co. Ltd(Tianjin, China)
Sulphuric acid	GR	Kermel Chemical Reagent Co. Ltd(Tianjin, China)
Hydrofluoric acid	GR	Kermel Chemical Reagent Co. Ltd(Tianjin, China)
Hydrogen peroxide	GR	Kermel Chemical Reagent Co. Ltd(Tianjin, China)
Ultrapure water	HPLC	Purified on a Milli-Q system supplied by Millipore(Bedford MA, USA)
Titanium	GR	National Center for Reference Materials
Lead	GR	National Center for Reference Materials
Chromium	GR	National Center for Reference Materials
Cadmium	GR	National Center for Reference Materials

2.2 Experimental instruments for detecting harmful chemical residues

The instrument information used in this experiment is shown in Table 2.

Table 2 Experimental instrument information table

<i>Instrument</i>	<i>Model</i>	<i>Manufacturer</i>
Electronic balance	BSA4202S-CW	Guangzhou Shenhua Biotechnology Co., Ltd.
Multiwave ECO	IN-WB24	Shandong Laiyin Optoelectronic Technology Co., Ltd.
Inductively coupled plasma atomic emission spectrometer	ICP-8000	Wuxi Chuangxiang Analytical Instrument Co., Ltd.
Milli-Q advantage	EDI	Liaoning Huamo Water Treatment Environmental Protection Equipment Co., Ltd.

2.3 Methods for detecting harmful chemical residues

After clarifying the materials, instruments, and basic operating procedures, Section 2 enters the stage of condition optimisation and result analysis. This part is carried out in sequence based on the ratio of digestion reagents, digestion temperature and time, and element detection spectral line screening. Each round of optimisation is based on the optimal conditions established in the previous round, ultimately forming a complete method parameter system, followed by method performance validation and actual sample testing.

2.3.1 Preparation of standard solution

For the four elements of titanium, lead, chromium, and cadmium, a calibrated 1.0 mL pipette was used to accurately transfer 1.0 mL of each standard stock solution to a 100 mL A-class volumetric flask (Hu et al., 2025; Du et al., 2025; Zhang et al., 2025; Sun et al., 2025). Subsequently, the solution was prepared by diluting with ultrapure water, adjusting the volume, and shaking well. Afterwards, it was used as the mother liquor and diluted with dilute nitric acid at a volume fraction of 3% to achieve standard solution concentrations of 0, 10, 20, 40, 80, and 100 µg/L. After preparation, it was stored at 4°C for future use (Yang and Wang, 2024; Jia et al., 2024).

2.3.2 Sample pretreatment

Firstly, cut the selected regenerated cellulose based food packaging material to be tested into small pieces, weigh 0.5 g of the small pieces and place them in a microwave digestion tank. Add concentrated nitric acid, hydrogen peroxide, and hydrofluoric acid in sequence, seal and dissolve according to the preset procedure. After digestion, heat and concentrate at 200°C until 1~2 mL remains (Luo et al., 2025; Wang et al., 2024; Ying et al., 2024; Wang et al., 2024). After cooling, transfer the digestion solution to a 10 mL stoppered colorimetric tube, wash the tank walls multiple times with ultrapure water, and combine the washings (Zhang et al., 2023; Fu et al., 2023; Yan et al., 2023). Finally, dilute to 10 mL, shake well and let stand. After the solution is completely clear, filter it through a 0.45µm filter membrane. The obtained test solution can be used for

determining the content of each element by ICP-AES method. The parameter table of microwave digestion program is shown in Table 3.

Table 3 Parameter table of microwave digestion program

<i>Step</i>	<i>Time/min</i>	<i>Temperature/°C</i>	<i>Power/W</i>
1	5	90	1000
2	10	140	1000
3	15	180	1000
4	20	200	1000

2.3.3 Instrument condition settings

The inductively coupled plasma atomic emission spectrometer (ICP-AES) was used for analysis and determination, and the specific working parameters are shown in Table 4.

Table 4 Instrument parameter information setting table

<i>Indicator</i>	<i>Parameter value</i>
RF power/kW	1.1
Plasma gas flow rate/L·min ⁻¹	19
Auxiliary airflow rate/L·min ⁻¹	0.5
Nebuliser pressure/MPa	0.2
Sample injection flow rate/mL·min ⁻¹	1.4
Signal integration time/s	10

3 Results and analysis of harmful chemical residue detection

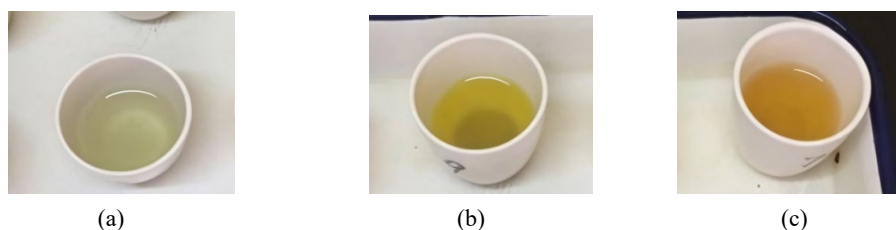
On the basis of optimising the digestion conditions and analysis spectral lines, the performance of the method was systematically validated from two dimensions: linear range and detection limit, recovery rate and precision. The established method was applied to the detection of actual samples on the market to test its applicability in real matrices. Finally, through horizontal comparison with current national standards and literature methods, the technical advantages of this method were further clarified.

3.1 Determination of digestion solution ratio

Given the complex composition of regenerated cellulose materials, in addition to the main cellulose, there may also be a small amount of inorganic fillers, coatings, or processing aids present. Using the HNO₃-H₂O₂ binary system alone cannot completely decompose all matrix components, resulting in incomplete digestion or low element recovery rates. Therefore, this study introduces hydrofluoric acid (HF) to investigate the digestion effect of the HNO₃-HF-H₂O₂ ternary mixture system. Select typical commercially available regenerated cellulose based food packaging materials as the research object. First, fix the total volume of the digestion solution to 8 mL. Based on the preliminary experiment, set different volume ratio combinations of HNO₃, HF, and H₂O₂

reagents at 5 : 1 : 2, 5 : 2 : 1, and 6 : 1 : 1, respectively. Under the microwave digestion program in Table 3, each group of samples was digested. After digestion was completed, the digestion solution was transferred and diluted. By observing the clarity, colour, and precipitation amount of the digestion solution, the digestion effect was comprehensively evaluated, and the results are shown in Figure 1.

Figure 1 Comparison of different digestion solution ratios: (a) when the ratio is 5 : 1 : 2; (b) when the ratio is 5 : 2 : 1 and (c) when the ratio is 6 : 1 : 1 (see online version for colours)

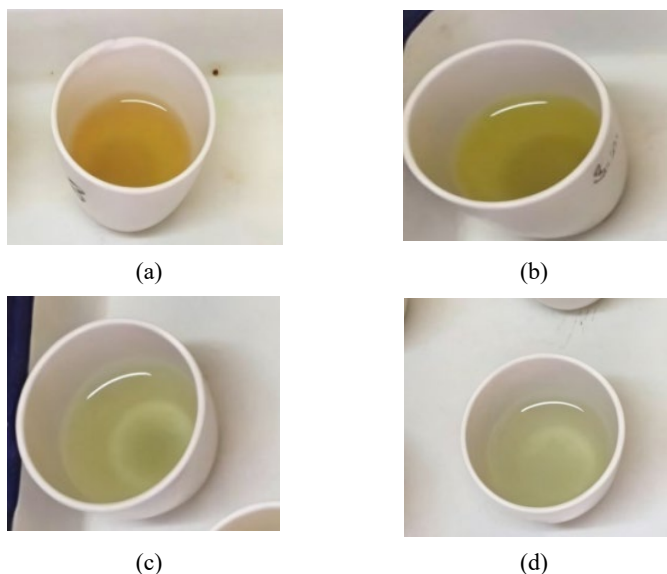


As shown in Figure 1, there are differences in the digestion effect of ternary systems with different proportions. When the ratio of HNO_3 : HF : H_2O_2 is 5 : 1 : 2, the sample is most thoroughly digested, and the digestion solution becomes colourless and transparent after cooling to a certain volume. There is no obvious precipitation after standing. At this ratio, nitric acid provides the main oxidising and acidic environment, sufficient hydrogen peroxide further enhances the oxidative decomposition of the organic matrix, and the addition of an appropriate amount of hydrofluoric acid effectively solves the possible problem of silicate substance encapsulation, ensuring the complete release of the target element. In contrast, other proportion solutions appear slightly yellow or accompanied by a small amount of suspension and precipitation. Therefore, considering the completeness of digestion, the safety of reagents, and the adaptability of the final solution, HNO_3 :HF: H_2O_2 =5 : 1 : 2(V/V) is determined as the optimal reagent ratio for microwave digestion of regenerated cellulose based food packaging materials. The selection of this ratio has clear chemical basis. Regenerated cellulose materials may introduce silicate fillers or silicon containing coatings during processing. Although the conventional HNO - H_2O_2 system can effectively oxidise organic matrices, it cannot destroy the lattice structure of silicates, resulting in incomplete release of some target elements due to encapsulation. The introduction of HF can selectively dissolve silicates and release the metal elements encapsulated within them. The determination of the volume ratio of 5 : 1 : 2 is based on the following three considerations: HNO_3 has the highest volume ratio to ensure sufficient oxidation of organic matter; The proportion of HF is moderate, which is sufficient to destroy the silicate structure while avoiding the safety risks and corrosion of the instrument caused by excessive use; H_2O_2 can generate highly active hydroxyl radicals under heating conditions, and synergistically enhance oxidation efficiency with HNO_3 . Comparative experiments show that under this ratio, the digestion solution is colourless, transparent, and free of precipitation, while the 5 : 2 : 1 ratio may cause the digestion solution to appear slightly yellow due to the high HF ratio, indicating the possibility of excessive corrosion or side reactions; 6: Due to insufficient HF, some samples still have trace amounts of precipitation in a 1 : 1 ratio. Therefore, the 5 : 1 : 2 ratio achieves the optimal balance between complete digestion, reagent safety, and subsequent instrument compatibility.

3.2 Determination of digestion temperature and time

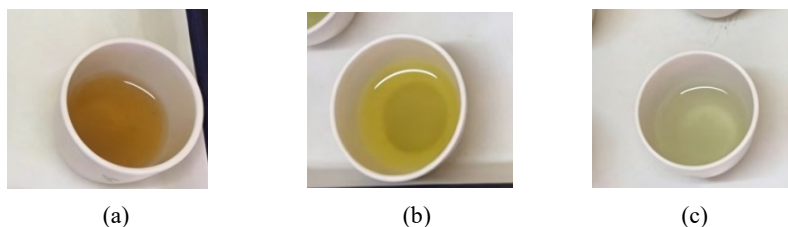
After determining the digestion reagent system, it is necessary to further optimise the key parameters in the microwave digestion program – temperature and time – to ensure that the digestion process is both efficient and safe. The core of this study is to determine the final digestion temperature and the constant temperature time at that temperature. Therefore, the power is fixed at 1000 W. Then, the constant temperature time is set to 20 min, and the digestion effects are compared at temperatures of 90°C, 140°C, 180°C, and 200°C. The results are shown in Figure 2.

Figure 2 Comparison of different temperatures: (a) when the temperature is 90°C; (b) when the temperature is 140°C; (c) when the temperature is 180°C and (d) when the temperature is 200°C (see online version for colours)



The experiment found that at 200°C, the sample was digested more thoroughly and the clarity of the digestion solution was higher, indicating that higher temperatures are beneficial for the complete decomposition of such materials. Subsequently, after determining the final temperature to be 200°C, the focus was on optimising the constant temperature time. Three levels of 10, 15, and 20 min were set for comparison, and the results are shown in Figure 3.

Figure 3 Comparison at different times: (a) when the time is 10 min; (b) when the time is 15 min and (c) when the time is 20 min (see online version for colours)



According to the analysis of Figure 3, when the constant temperature time reached 20 min, all the regenerated cellulose samples tested achieved complete digestion, and the resulting digestion solution was clear and transparent, without visible particles or obvious colour. In contrast, if the constant temperature time is insufficient, it may lead to incomplete digestion of some samples, thereby affecting the accuracy and reproducibility of the analysis results. Based on comprehensive experimental data, the optimal conditions for microwave digestion of regenerated cellulose based food packaging materials were determined to be a temperature of 200°C and a constant temperature of 20 min.

3.3 *Element detection spectral line determination*

In the same sample matrix, multiple emission lines of the same element by ICP-AES may be subject to varying degrees of spectral interference or background influence. To obtain accurate and stable measurement results, it is necessary to screen the optimal analytical spectral lines for each element to be tested in the solution system after the digestion of regenerated cellulose based food packaging materials. In this study, we first examined the spectral line intensity, signal-to-noise ratio, and possible spectral line overlap or matrix interference to select detection spectral lines. Based on this, we further verified the reliability of spectral line selection through spiked recovery experiments, and the results are shown in Table 5.

Table 5 Element detection spectral line information table

<i>Element</i>	<i>Wavelength</i>	<i>RSD/%</i>
Ti	334.941	95.3
	337.281	84.4
	336.118	88.9
Pb	220.353	94.3
	252.183	79.4
	280.718	79.5
Cr	206.783	91.03
	267.783	84.93
	284.831	68.94
Cd	205.382	78.93
	214.839	93.13
	228.934	82.42

According to the data in Table 5, the selected spectral line wavelengths for titanium, lead, chromium, and cadmium in this study are 334.941 nm, 220.353 nm, 206.783 nm, and 214.839 nm, respectively. The above spectral lines have low background and moderate sensitivity at the selected wavelength, and there is no obvious spectral interference between them. At the same time, the instrument's built-in spectral background correction function further deducts the background influence, which can meet the requirements of accurate determination of multiple elements in regenerated cellulose matrix at the same time. The scientific basis for this spectral line combination is reflected in the following aspects:

Ti element has the highest signal-to-noise ratio at 334.941 nm, with spectral line intensities about 12.9% and 7.2% higher than 337.281 nm and 336.118 nm, respectively. There are no characteristic interference peaks from common coexisting elements such as Fe, Ca, and Mg in the regenerated cellulose digestion solution near this wavelength. Although the sensitivity of the 220.353 nm spectral line of Pb is slightly lower than that of 217.000 nm, the latter is susceptible to spectral overlap interference from Al and Fe, while Al is common in regenerated cellulose materials. Therefore, the 220.353 nm spectral line with stronger anti-interference ability is selected as the analysis line. The 206.783 nm spectral line of Cr exhibits better stability at low concentrations, with a relative standard deviation of 91.03%, significantly higher than the 68.94% at 284.831 nm, indicating better repeatability of this spectral line in complex matrices. The 214.839 nm spectral line of Cd is less affected by the interference of Fe and Zn spectral lines compared to 205.382 nm and 228.934 nm, and the background noise is lower at this wavelength. In addition, all selected spectral lines in this study were validated through spiked recovery experiments, with recovery rates ranging from 83% to 108%, confirming the accuracy and feasibility of spectral line selection in actual regenerated cellulose matrices. The instrument background correction function further compensates for residual baseline drift, ensuring the reliability of data when multiple elements are measured simultaneously.

3.4 Performance verification of detection methods for harmful chemical residues

Using the established method, actual testing was conducted on 5 commercially available regenerated cellulose based food packaging materials, while setting up reagent blanks for control. The results are shown in Table 6.

Table 6 Information on actual sample testing results

Sample number	Ti/mg·kg ⁻¹	Pb/mg·kg ⁻¹	Cr/mg·kg ⁻¹	Cd/mg·kg ⁻¹
1	ND	0.15	ND	0.32
2	0.05	0.08	ND	0.21
3	ND	0.18	0.01	ND
4	0.11	0.22	0.45	ND
5	0.32	0.09	0.01	0.07

ND indicates not detected.

As shown in Table 6, titanium (Ti), lead (Pb), chromium (Cr), and cadmium (Cd) were detected to varying degrees in actual samples. The detection frequency of lead and cadmium is relatively high, with content ranges of 0.08~0.22 mg·kg⁻¹ and 0.07~0.32 mg·kg⁻¹, respectively; Among various elements, titanium only appears in some samples, with the highest element content being only 0.32 mg·kg⁻¹; The detected content of chromium is relatively low, ranging from 0.01 mg·kg⁻¹ to 0.45 mg·kg⁻¹. The content of each element in all samples did not exceed the limit requirements stipulated by relevant national food safety standards, indicating that the migration risk of harmful elements in the tested packaging materials is generally controllable. This method is suitable for rapid screening and actual detection of multiple elements in regenerated cellulose based food

packaging materials. Based on the experimental data, the main factors affecting the detection accuracy are systematically analysed, as follows:

The interference of sample matrix is the primary factor leading to measurement deviation. Coexisting elements such as calcium, magnesium, iron and aluminium, which are ubiquitous in regenerated cellulose materials, may cause spectral overlap interference or physical matrix effect during ICP-AES determination. Specifically, near the 220.353 nm spectral line of Pb element, the 220.467 nm spectral line of Al may partially overlap. Although the spectral line screened this time has avoided this interference to the greatest extent, when the Al concentration in the sample exceeds $10 \text{ mg} \cdot \text{kg}^{-2}$, it is still necessary to monitor the change of background signal. The 206.783 nm spectral line of Cr is easily disturbed by the 206.799 nm spectral line of Fe, which may lead to the higher measured value of Cr by about 3–8% in samples with high Fe content. In addition, the porous hydrophilic structure of regenerated cellulose may produce incomplete carbonised products during the digestion process, and these organic residues will change the viscosity of the solution, affect the atomisation efficiency and aerosol transmission, thus reducing the signal stability. In view of the above interference, this study adopts the following improvement measures:

Firstly, the high-resolution mode and three-point background correction function are enabled in the instrument parameter setting, and the background signals on both sides of each analytical spectral line are collected and deducted respectively, thus effectively suppressing spectral overlapping interference;

Secondly, the internal standard element yttrium with the same concentration was added to both the standard solution and the sample solution, and the final concentration was $1 \text{ mg} \cdot \text{L}^{-1}$ to correct the fluctuation of atomisation and excitation efficiency caused by the difference of physical properties of the solutions.

Thirdly, for the special samples with significantly high Fe and Al contents, the standard addition method is used to replace the external standard method for quantification, so as to eliminate the systematic error caused by the difference of matrix matching;

Fourthly, after the digestion procedure is completed, the acid removal step is added. When the digestion solution is concentrated to about 0.5 mL, 1 mL of HNO₃ is added for repeated concentration, so as to remove the residual HF and organic degradation products to the greatest extent and reduce the chemical interference to the subsequent determination.

Through the above improvements, the recoveries of Pb and Cr in typical spiked samples increased from 78.5% and 81.2% before optimisation to 89.3% and 93.2% respectively, and the relative standard deviations decreased from 9.5% and 10.1% to 4.8% and 5.4%, which verified the effectiveness of the improvement measures. The method system has good inhibition ability to common interference factors in regenerated cellulose matrix, and can meet the requirements of accurate determination of trace harmful elements in actual samples.

3.4.1 Detection limit and linear range of harmful chemical residues

A dilute nitric acid solution with a volume fraction concentration of 3% was used as the dilution medium to prepare standard curve solutions of different concentrations, with concentration gradients set at 0.01, 0.05, 0.1, 1, 5, 10, and $20 \text{ mg} \cdot \text{L}^{-1}$. Based on this, the blank solution and various concentration standard solutions were measured separately,

and the linear regression equations, correlation coefficients, detection limits, and quantification limits of each element were obtained as shown in Table 7.

Table 7 Linear equations, detection limits, and determination limits for each element

<i>Element</i>	<i>Linear equation</i>	<i>Correlation coefficient/r</i>	<i>Detection limit/$\mu\text{g}\cdot\text{kg}^{-1}$</i>	<i>Quantitative limit/$\mu\text{g}\cdot\text{kg}^{-1}$</i>
Ti	$Y = 9.7680x + 0.028$	0.9996	3.81	12.9
Pb	$Y = 1.983X + 0.025$	0.9998	10.82	36.22
Cr	$Y = 11.927X + 0.087$	0.9989	12.94	43.19
Cd	$Y = 56.072X - 0.785$	0.9996	1.96	6.53

Analysis of the data in Table 7 shows that all target elements exhibit good linear relationships within the range of 0.01~20 $\text{mg}\cdot\text{L}^{-1}$, with correlation coefficients (r) greater than 0.989.

3.4.2 Recovery rate and precision of detection methods for harmful chemical residues

To evaluate the accuracy and repeatability of this method, this study selected three different sources of regenerated cellulose food packaging material samples to conduct spiked recovery experiments at three concentration levels. The final results are shown in Table 8.

Table 8 Average yield and precision of microwave digestion ($n = 6$)

<i>Elements to be tested</i>	<i>Add concentration/$\text{mg}\cdot\text{kg}^{-1}$</i>	<i>Average recovery rate/%</i>	<i>RSD/%</i>
Ti	0.02	90.37	6.71
	0.10	108.32	5.28
	0.50	97.39	3.81
Pb	0.02	83.19	5.78
	0.10	89.29	4.12
	0.50	93.11	4.78
Cr	0.02	88.41	7.41
	0.10	89.29	5.37
	0.50	93.21	2.12
Cd	0.02	85.21	6.31
	0.10	87.23	4.42
	0.50	90.18	2.17

As shown in Table 8, the average spiked recovery rates of each tested element in various matrices ranged from 83.19% to 108.32%, with relative standard deviations (RSD) in the range of 2.12–7.41%. This result indicates that the established method has ideal accuracy and precision, and can meet the analysis requirements of multi-element synchronous detection in actual samples.

In order to further verify the progressiveness and practical value of the proposed method, this research method is compared horizontally with the current national standards and literature reporting methods. The specific results are shown in Table 9.

Table 9 Comparison of our research methods with national standards and literature methods

<i>Comparison items</i>	<i>Proposed method</i>	<i>GB 31604.49–2016</i>	<i>Literature reporting method</i>
Detection technology	ICP-AES	ICP-MS (Unique Designated Method for Elemental Determination of Paper Products)	ICP-AES/ICP-MS
Pre processing system	HNO ₃ -HF-H ₂ O ₂ ternary system	Not specifically optimised for regenerated cellulose matrix	Mostly HNO ₃ -H ₂ O ₂ system
Applicable matrix	Regenerated cellulose food packaging materials	Paper products, cork stoppers, etc.	Multiple food packaging materials
Detection limit/ μg·kg ⁻¹	1.96~12.94	ICP-MS method can reach ppb level	Equivalent to this study
Additive recovery rate/%	83.19~108.32	Within the scope of standard requirements	92 ± 14 (ICP-MS)
Precision RSD/%	2.12~7.41	—	—
Simultaneous determination of multiple elements	Support (Ti, Pb, Cr, Cd)	Support (As, Cd, Cr, Pb, etc.)	Support
Instrument cost and maintenance	Lower	Higher (ICP-MS)	Higher
Consistency with current standards	Fill the gap in the detection of regenerated cellulose matrix	Not specifically revised for regenerated cellulose materials	Not targeting regenerated cellulose materials

GB 31604.49-2016, as the current national standard for the determination of arsenic, cadmium, chromium, and lead in food contact materials and products, adopts inductively coupled plasma mass spectrometry as the only designated method for element determination in paper products. Inductively coupled plasma emission spectroscopy is only applicable for the determination of migration volume, and the pre-treatment method of this standard does not specify the special digestion requirements for regenerated cellulose matrix. In contrast, this research method focuses on the possible presence of silicate fillers or coatings in regenerated cellulose materials, and introduces HF to construct a ternary digestion system based on the HNO₃-H₂O₂ system. The spiked recovery rates of Ti, Pb, Cr, and Cd elements reach 83.19–108.32%, which is significantly better than the recovery rates reported in literature using only the HNO₃-H₂O₂ system for the digestion of silicon containing substrates. In terms of detection limit, the detection limit of each element in this study is 1.96 to 12.94 μg·kg⁻¹, which is on the same order of magnitude as the detection limit of inductively coupled plasma mass

spectrometry in GB 31604.49-2016. However, this method uses an inductively coupled plasma emission spectrometer for determination, which has lower instrument operating costs, simpler daily maintenance, and is more suitable for routine screening of bulk samples. In terms of digestion efficiency, the 200°C constant temperature 20 min digestion program established in this study is superior to the microwave digestion conditions reported by Perring et al. This study used the HNO₃-HF-H₂O₂ system to digest various food packaging materials, but did not specifically optimise the silicon containing regenerated cellulose matrix. In terms of standard applicability, GB 4806.8-2022 “National Food Safety Standard for Food Contact Paper and Paperboard Materials and Products” has removed clauses that are not applicable to regenerated cellulose materials and officially included regenerated cellulose materials in the scope of standard management. However, the corresponding supporting testing method standards have not been revised specifically. The establishment of this research method can fill the methodological gap in this field. In addition, existing industry standards such as SN/T 2900-2011 and SN/T 3551-2013 focus on the detection of organic substances or specific single elements, and have not yet covered the inductively coupled plasma emission spectroscopy scheme for simultaneous determination of multiple elements such as Ti, Pb, Cr, Cd, etc. In summary, this method has comprehensive advantages over current national and industry standards in terms of targeted digestion systems, multi-element synchronous analysis capabilities, operating costs, and operational convenience. It can provide a reliable technical solution for efficient screening of harmful elements in regenerated cellulose food packaging materials.

In summary, this study aims to address the technical difficulties of the complex matrix of regenerated cellulose food packaging materials, the difficulty of complete decomposition in conventional digestion systems, and the prominent interference of spectral lines in multi-element synchronous detection. The detection method is systematically constructed from two levels: sample pretreatment optimisation and instrument analysis condition screening. In the pre-processing stage, an innovative HNO₃-HF-H₂O₂ ternary mixed digestion system was introduced, and three volume ratios of 5:1:2, 5:2:1, and 6:1:1 were compared and optimised to solve the problem of incomplete element release caused by silicate fillers or coatings; Simultaneously set four temperature levels of 90, 140, 180, and 200°C, as well as three constant temperature time levels of 10, 15, and 20 min, to systematically investigate the effects of digestion temperature and time on the degree of sample decomposition, in order to establish the optimal program for complete digestion of regenerated cellulose matrix. In the instrument analysis stage, in order to address the issues of spectral line overlap and matrix interference in the simultaneous determination of multiple elements by ICP-AES, spectral line intensity, signal-to-noise ratio, and spiked recovery rate were used as evaluation indicators to screen the best analysis spectral line with high signal-to-noise ratio and low interference for Ti, Pb, Cr, and Cd elements from multiple candidate spectral lines. The instrument background correction function was used to further reduce the influence of matrix. In the method performance verification stage, the sensitivity, accuracy, and repeatability of the method were systematically evaluated through indicators such as linear range, detection limit, quantification limit, spiked recovery rate, and precision. The established method was applied to actual testing of five commercially available regenerated cellulose food packaging materials to verify their applicability in complex matrices. The core innovation of the above technical route lies in applying the ternary digestion system to the pretreatment of regenerated cellulose based packaging materials,

effectively solving the problem of incomplete digestion caused by its porous hydrophilic matrix. A spectral line screening strategy combining full spectrum scanning and spiked recovery verification is carried out for this specific matrix, providing a systematic solution to suppress complex spectral interference.

Further analysis of the experimental results can summarise the following detection rules and their inherent correlation with the adopted methods, as follows:

Firstly, the completeness of digestion directly determines the efficiency of element release and measurement accuracy. Experimental data shows that only when the digestion solution is completely colourless and transparent, and there is no precipitation when left to stand, can the spiked recovery rates of the four target elements be stably maintained at over 85%; If the digestion solution appears slightly yellow or contains trace amounts of suspended solids, the recovery rates of Ti and Cr decrease by 12–18% and 8–15%, respectively, indicating that incomplete digestion has a more significant impact on high melting point elements.

Secondly, the selection of spectral lines is closely related to the composition of the matrix. The average contents of Ca, Mg, Fe, and Al in the regenerated cellulose sample are $126 \text{ mg}\cdot\text{L}^{-1}$, $84 \text{ mg}\cdot\text{L}^{-1}$, $37 \text{ mg}\cdot\text{L}^{-1}$, and $52 \text{ mg}\cdot\text{L}^{-1}$, respectively. The distribution of the coexisting elements directly affects the signal-to-noise ratio and anti-interference ability of the candidate spectral lines. The experimental verification shows that the four selected spectral lines can maintain a relative standard deviation of less than 6% under the fluctuation of the matrix, reflecting the good compatibility between the spectral lines and the matrix.

Thirdly, there is a strict linear relationship between the concentration of the target element and the detection signal, with correlation coefficients greater than 0.9989. This linear pattern is not affected by differences in sample source and matrix, indicating that the method has stable quantitative ability in the concentration range of $0.01\text{--}20 \text{ mg}\cdot\text{L}^{-1}$. Fourthly, the intrinsic correlation between the detection results and the adopted method is manifested as follows: the ratio of $\text{HNO}\text{--}\text{HF}\text{--}\text{H}_2\text{O}_2$ in the pre-processing stage is 5 : 1 : 2 and the digestion program is maintained at 200°C for 20 min, which determines the upper limit of element extraction efficiency; The spectral line screening and background correction strategies in the instrument analysis process determine the signal-to-noise ratio and anti-interference ability of signal pickup; Both factors jointly determine the accuracy, precision, and detection limit of the final test results.

The main innovation and research contribution of this work lies in the first application of the $\text{HNO}\text{--}\text{HF}\text{--}\text{H}_2\text{O}_2$ ternary digestion system to the pretreatment of regenerated cellulose food packaging materials, which solves the problem of incomplete element release caused by silicate encapsulation; A spectral line screening strategy combining full spectrum scanning and spiked recovery verification for this specific matrix has been established, effectively suppressing complex spectral interference; The system reveals quantitative correlation rules between dissolution parameters, spectral line selection, and detection performance, providing a generalisable methodological paradigm for multi-element synchronous analysis in similar complex matrix materials.

4 Conclusion

This study aims to address the technical difficulties of complex matrix of regenerated cellulose food packaging materials, difficult complete decomposition of conventional

digestion systems, and prominent spectral line interference in multi-element synchronous detection. A set of harmful chemical residue detection methods based on microwave digestion ICP-AES was systematically constructed, and the following original discoveries and method contributions were made: for the first time, the HNO_3 -HF- H_2O_2 ternary mixed digestion system was applied to the pretreatment of regenerated cellulose based food packaging materials. By comparing three volume ratios of 5 : 1 : 2, 5:2:1, and 6:1:1, 5 : 1 : 2 was determined as the optimal reagent ratio. This system effectively solves the problem of incomplete element release caused by silicate fillers or coatings, making the digestion solution colourless, transparent, and precipitation free, significantly improving Ti and Pb. Extraction efficiency of elements such as Cr and Cd; In the optimisation of the digestion program, four temperature levels of 90, 140, 180, and 200°C and three constant temperature time levels of 10, 15, and 20 min were set to establish a constant temperature of 200°C for 20 min as the optimal digestion condition. This ensures complete digestion while balancing operational safety and reasonable consumption of acid reagents; in the analysis of spectral line screening, a system optimisation strategy combining full spectrum scanning and spiked recovery verification was carried out for regenerated cellulose matrix for the first time. The spectral line intensity, signal-to-noise ratio, and spiked recovery rate were used as evaluation indicators to screen multiple candidate spectral lines of various elements, and Ti was 334.941 nm, Pb was 220.353 nm, Cr was 206.783 nm, and Cd was 214.839 nm as the optimal analysis spectral line. The instrument background correction function was also used to effectively suppress matrix spectral interference; Methodological validation showed that the linear relationship between each element was good within the range of 0.01~20mg·L⁻¹, with correlation coefficients greater than 0.9989. The detection limit was as low as 1.96~12.94µg·kg⁻¹, and the quantification limit was 6.53~43.19µg·kg⁻¹. The sensitivity was significantly better than conventional empirical line selection schemes, and the spiked recovery rate was 83.19%~108.32%, with a relative standard deviation of 2.12%~7.41%. The accuracy and precision met the requirements for trace analysis of multiple elements in complex matrices; The established method was applied to conduct actual testing on five commercially available regenerated cellulose food packaging materials, and Ti, Pb, Cr, and Cd were all detected to varying degrees but below the national food safety standard limits, confirming the practical applicability of the method. The method developed in this study has significant advantages in digestion efficiency, anti-interference ability, and multi-element synchronous analysis performance, which can provide reliable technical tools for daily monitoring, risk assessment, and revision of green packaging standards for harmful chemical residues in regenerated cellulose food packaging materials.

Conflicts of interest

All authors declare that they have no conflicts of interest.

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