Int. J. Electrochem. Sci., 16 **(2021)** Article ID: 210652, doi: 10.20964/2021.06.06

International Journal of
ELECTROCHEMICAL
SCIENCE
www.electrochemsci.org

Simultaneous Determination of Seven Carbamate Pesticide Residues in Vegetable by Capillary Electrophoresis with Solid Phase Microextraction

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Received: 19 January 2021 / *Accepted:* 26 March 2021 / *Published:* 30 April 2021

An efficient method for simultaneous determination of seven carbamate pesticides in vegetable was established based on purifing and enriching by solid phase microextraction, separating by capillary electrophoresis (CE) and detecting by column end electrochemiluminescence (ECL). The effects of acidity, time, temperature, ionic strength and eluent on the recovery of seven pesticides in sample solutions were investigated. The best purification and enrichment parameters of 0.2 g hollow fiber in 10.0 mL extraction solution are: pH value 6, extraction time 35 min, extraction temperature 50℃, NaCl 1.5 g, and 2.0 mL acetonitrile (containing 5% formic acid) as eluent. The effects of acidity, salt concentration, additive concentration and separation voltage on the resolution of seven pesticides in separation medium were investigated. The phosphate buffer solution (PBS) containing 45 mmol/L NaCl and 25 mmol/L cyclodextrin was used as separation solution of CE. The pH of PBS is 6.5. The separation voltage is 17 kV. There are good linear relationships between the mass concentrations of seven carbamate pesticides and their ECL intensities. Their detection limits were 0.1-0.5 μg/L. The recoveries of standard addition for vegetable samples were 86.1%-115.8%. The method is rapid and accurate for the determination of carbamate pesticides in vegetables.

Keywords: Carbamate pesticide, Vegetable, Solid phase microextraction, Capillary electrophoresis

1. INTRODUCTION

Generally, only about 30% of the pesticides applied in farmland are used by crops, and the rest enter the soil or diffuse into the atmosphere, causing serious pesticide pollution. With the increasing range and amount of pesticide use, pesticide residues in the soil finally enter the human body through the food chain and endanger human health [1-4]. Carbamates are a kind of pesticides with broad-spectrum biological activity. It can kill insects by inhibiting the activity of acetylcholinesterase. In

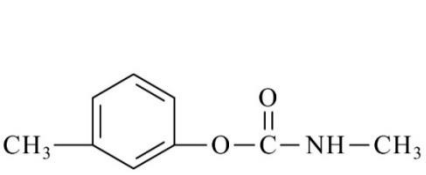
recent years, these pesticides have been widely used in agriculture, forestry and animal husbandry due to their fast decomposition, short residue period and low bioaccumulation. At present, more than 1000 carbamate pesticides are available. But the most commonly used are metolcarb (MTC), carbaryl (CBY), methomyl (MTM), aldicarb (ADC), carbofuran (CBF), isoprocarb (IPC) and pirimicarb (PMC). However, their chemical properties, metabolic processes and metabolites are not well understood. It is necessary to do in-depth research on the environmental and health problems caused by its migration in the ecological environment. Studies have found that carbamate pesticides can cause carcinogenesis in rats and hamsters by oral administration, injection or application to the skin [5-7]. In view of this, it is particularly important to find an ideal analysis method for simultaneous determination of multiple carbamate pesticides.

The detection methods of carbamate pesticide residues mainly include fluorescence [8-12], electrochemistry [13-15], biosensor [16-19], gas chromatography (GC) - mass spectrometry (MS) [20-22], liquid chromatography (LC) [23-28], LC-MS [29-31], LC-ultraviolet (UV) [32], surface-enhanced raman spectroscopy [33], thin-layer chromatography (TLC) [34], and colorimetry [35,36]. With the improvement of people's living standards, more and more food needs to be detected, and the requirements for rapid analysis are higher and higher. Therefore, it is very important to establish a fast and convenient detection method for simultaneous determination of various carbamate pesticide residues in different foods.

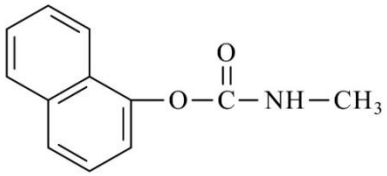
Capillary electrophoresis (CE) is an efficient separation method for trace substances in many fields. Electrochemiluminescence (ECL) based on tris (2,2'-bipyridyl) ruthenium (II) ($Ru(bpy)_3^{2+}$) is an attractive analytical method for organic amines owing to its inherent high sensitivity, high selectivity and high stability. The combination of CE and ECL has been widely used in the analysis of organic amine drugs [37-40], organic amine antibiotics [40-44] and organic amine pesticide residues [45-47] in many samples from different fields. Most carbamate pesticides contain secondary amine group or tertiary amino group (as shown in Figure 1). They can obviously enhance the ECL signal of $Ru(bpy)_3^{2+}$. Therefore, CE-ECL may be a good method for simultaneous separation and analysis of carbamate pesticides.

Sample pretreatment is the most time-consuming and laborious work in the analysis process. Solid phase microextraction (SPME) is a sample pretreatment technique which is very popular with analytical workers. It integrates sampling, extraction and concentration. Using this technology can greatly accelerate the speed of analysis and detection. It has been widely used in sample pretreatment of environmental [48-50], food [51-55] and pharmaceutical industries [56-59].

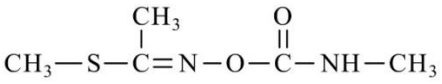
Vegetables are the necessary food materials in our daily life. Pesticide residues in vegetables have a great impact on people's health. In this paper, carbamate pesticides remaining in vegetable samples, such as cucumber, tomato, cabbage, celery, eggplant and leek, were purified and enriched by SPME technology. Then seven carbamate pesticides, such as MTC, CBY, MTM, ADC, CBF, IPC and PMC, were separated and detected simultaneously by CE-ECL. The results show that the present method is sensitive and reliable for the simultaneous determination of the seven carbamate pesticides in vegetables.



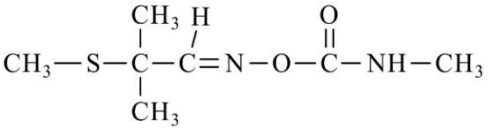
Metolcarb



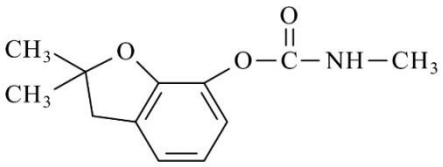
Carbaryl



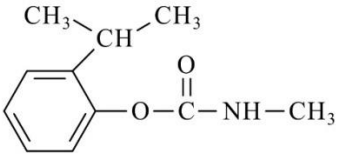
Methomyl



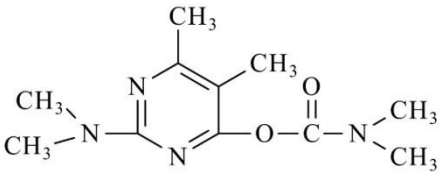
Aldicarb



Carbofuran



Isoprocarb



Pirimicarb

Figure 1. Molecular structures of seven carbamate pesticides.

2. EXPERIMENTAL

2.1. Materials and chemicals

Tris (2,2'-bipyridyl) ruthenium (II) dichloride hexahydrate ($Ru(bpy)_3Cl_2 \cdot 6H_2O$) was purchased from Alfa Aesar (Johnson Matthey, USA). Standard substances of Metolcarb (MTC), carbaryl (CBY), methomyl (MTM), aldicarb (ADC), carbofuran (CBF), isoprocarb (IPC) and pirimicarb (PMC) were purchased from National Institutes for Food and Drug Control (Beijing, China). Sodium hydroxide (NaOH), hydrochloric acid (HCl), disodium hydrogen phosphate (Na_2HPO_4), sodium dihydrogen phosphate (NaH_2PO_4), methanol, acetonitrile, formic acid, methyl orthosilicate, sodium chloride (NaCl), cyclodextrin, pyrrolidone, tween 40 and isopropanol were purchased from Beijing Chemical Reagent Company (Beijing, China). Polypropylene hollow fiber (inner diameter 600 μm and micropore 0.3 μm) was purchased from Tianjin Film Technology Co., Ltd (Tianjin, China).

2.2. Apparatus and conditions

CE-ECL was performed on a MPI - B multi-parameter chemiluminescence analysis test system (Xi'an Remex analytical instruments Co., Ltd., Xi'an, China). Cyclic voltammetry and potentiostatic method were carried out in a three electrodes system with a platinum working electrode of 500 μm in diameter, an Ag/AgCl reference electrode of 300 μm in diameter and a platinum wire auxiliary electrode of 1 mm in diameter. Capillary (25μm x 40 cm) was rinsed respectively with 0.1 mol/L NaOH solution for 20 min, secondary distilled water for 10 min and running buffer for 15 min before use.

ECL conditions: Detection potential is 1.15 V (vs. Ag/AgCl). Concentration of $Ru(bpy)_3^{2+}$ is 6 mmol/L. Concentration of PBS (pH 6.8) in test cell is 40 mmol/L. See our previous work [38] for details.

2.3. Solid phase treatment

Adding methyl orthosilicate into methanol solution, adding 30% hydrochloric acid while stirring, aging at room temperature for 24 hours, and then completely hydrolyzing methyl orthosilicate into silica sol. Immerse polypropylene hollow fiber with a length of 5 mm in silica sol. After ultrasonic vibration at room temperature for 40 minutes, the hollow fiber was taken out of the sol and dried at 100℃ for later use. This process is similar to our previous work [60].

2.4. Solid phase microextraction

Vegetable samples, such as cucumber, tomato, Chinese cabbage, celery, eggplant, leek, etc., are homogenized and weighed 5.0 g, put into a 50 mL centrifuge tube with cover, add 5 mL acetonitrile, roll on a vortex oscillator for 1 min, add 5 mL methanol, ultrasonically oscillate for 20 min, add distilled water to make the solution volume reach 16 mL, and put into an ice water bath. Centrifuge at 5000 rpm for 5 minutes. Take 8 mL of supernatant as sample solution, put it into another 15 mL centrifuge tube with cover, add 1.5 g of sodium chloride, shake to completely dissolve the sodium chloride, adjust the pH value to 6.0, add distilled water to make the volume of sample solution 10 mL, raise the temperature to 50℃, immerse 0.2 g of hollow fiber in the solution, and perform ultrasonic extraction for 35 minutes. After extraction, the hollow fiber was transferred to a test tube, 2.0 mL acetonitrile (containing 5% formic acid) was added as eluent, and ultrasonic vibration was carried out for 5 minutes. Blow the solution dry with nitrogen, and add 0.5 mL methanol water solution (1:1) along the tube wall to dissolve the analyte. After passing through 0.22 μm microporous membrane, the filtrate was ready for use.

3. RESULTS AND DISCUSSION

3.1. Choice of SPME conditions

SPME conditions have a significant impact on the extraction of target analytes. In this part, the

influence of SPME conditions was studied with the recovery of seven carbamate pesticides as the investigation index.

3.1.1 Effect of pH

The pH of sample solution changes from 4 to 9 and the recovery of seven carbamate pesticides is shown in Figure 2. With the increase of pH value of sample solution, the recovery of seven pesticides increased first and then decreased, and reached the maximum when pH value was 6.0. The pH of sample solution used in SPME in literatures is mostly 5.0 to 6.5 [49-53, 56], which is consistent with our conclusion.

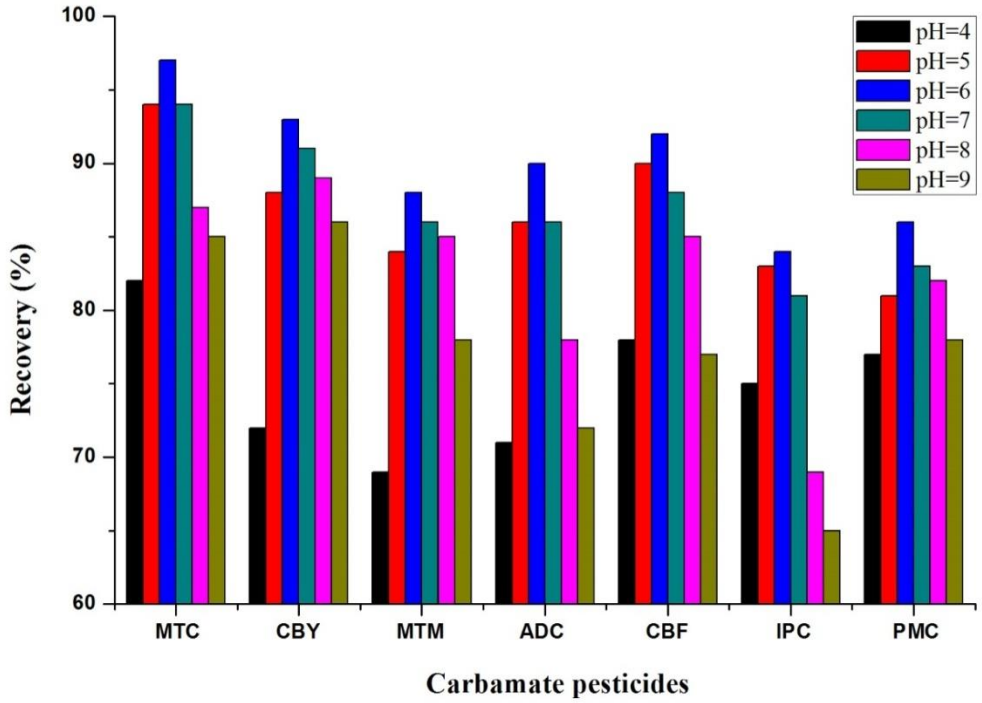


Figure 2. Effects of pH of 0.2 g hollow fiber in 10 mL sample solution on the recovery of seven carbamate pesticides under extraction time 35 min, extraction temperature 50℃ and NaCl 1.5 g.

3.1.2 Effect of extraction time

Figure 3 shows the effect of extraction time on the recovery rate of seven carbamate pesticides. With the extension of extraction time, the recovery of seven pesticides increased continuously, and reached a stable level after 35 min. Some people extract food ingredients for more than 50 minutes [58]. There was no significant difference in recovery after 35 min in our experiments.

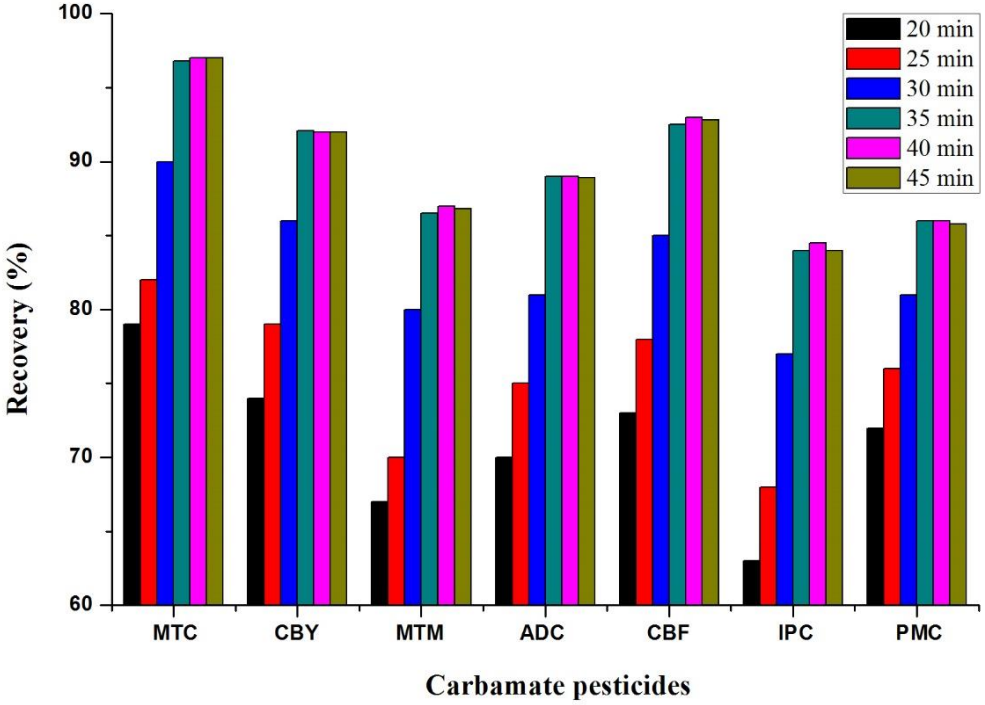


Figure 3. Effects of extraction time of 0.2 g hollow fiber in 10 mL sample solution on the recovery of seven carbamate pesticides under pH 6, extraction temperature 50℃ and NaCl 1.5 g.

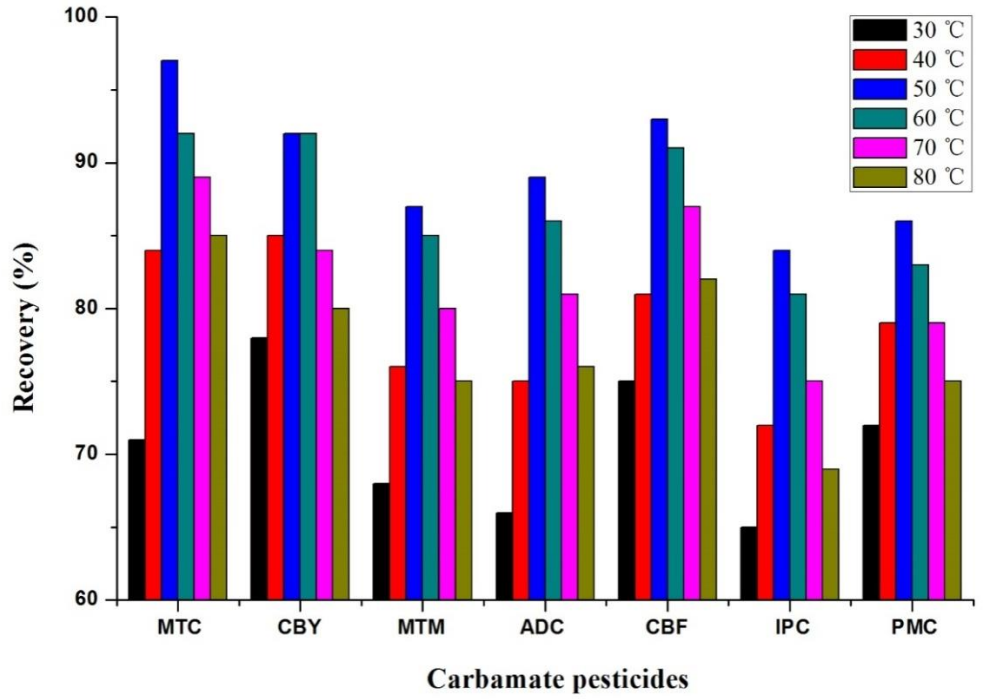


Figure 4. Effects of extraction temperature of 0.2 g hollow fiber in 10 mL sample solution on the recovery of seven carbamate pesticides under pH 6, extraction time 35 min and NaCl 1.5 g.

3.1.3 Effect of extraction temperature

This experiment investigated the effect of different extraction temperature on the extraction efficiency of seven carbamate pesticides, as shown in Figure 4. With the increase of extraction temperature, the recovery of seven pesticides first increased and then decreased, and reached the maximum at 50℃. In the literature, the extraction temperature is mostly about 50-60℃ [50-56]. But in our experiment, when the temperature is over 52℃, the extraction efficiency decreases obviously, and the recovery rate of some components drops to less than 80%. Therefore, our best extraction temperature is 50℃.

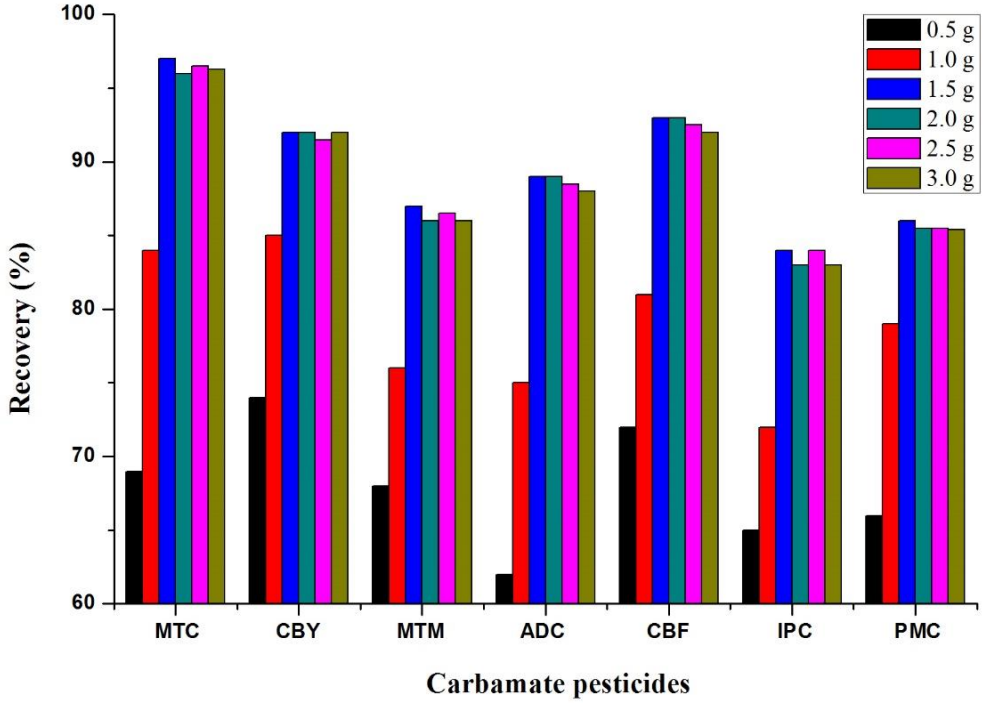


Figure 5. Effects of ionic strength of 0.2 g hollow fiber in 10 mL sample solution on the recovery under pH 6, extraction time 35 min and extraction temperature 50℃.

3.1.4 Effect of ionic strength

Due to the salting out effect, salt ions play a competitive role in the aqueous solution, which leads to the decrease of the dissolved organic matter concentration in the aqueous solution. Therefore, the salting out effect increases with the increase of ionic strength. The solubility of analyte in water decreased and the partition coefficient of analyte in fiber increased. Thus, the extraction efficiency can be improved. The effect of ionic strength on recovery was investigated by adding sodium chloride of different quality. As shown in Figure 5, with the increase of ionic strength, the recovery of seven carbamate pesticides increased, and reached the maximum when the concentration of sodium chloride was 1.5 g. The amounts of salt in literatures for SPME are mostly 1.0 to 2.0 [49-55, 57], which is

consistent with our conclusion.

3.1.5 Eluent of analyte

Methanol and acetonitrile are commonly used eluents in SPME in many literatures because of their strong solubility [48-56]. In this experiment, the effects of five eluents, (a) methanol, (b) methanol (containing 5% formic acid), (c) acetonitrile, (d) acetonitrile (containing 5% formic acid), and (e) methanol acetonitrile (1:1), on the recovery of seven carbamate pesticides were studied. The results are shown in Figure 6. The extraction efficiency of seven pesticides was good when acetonitrile (containing 5% formic acid) was used.

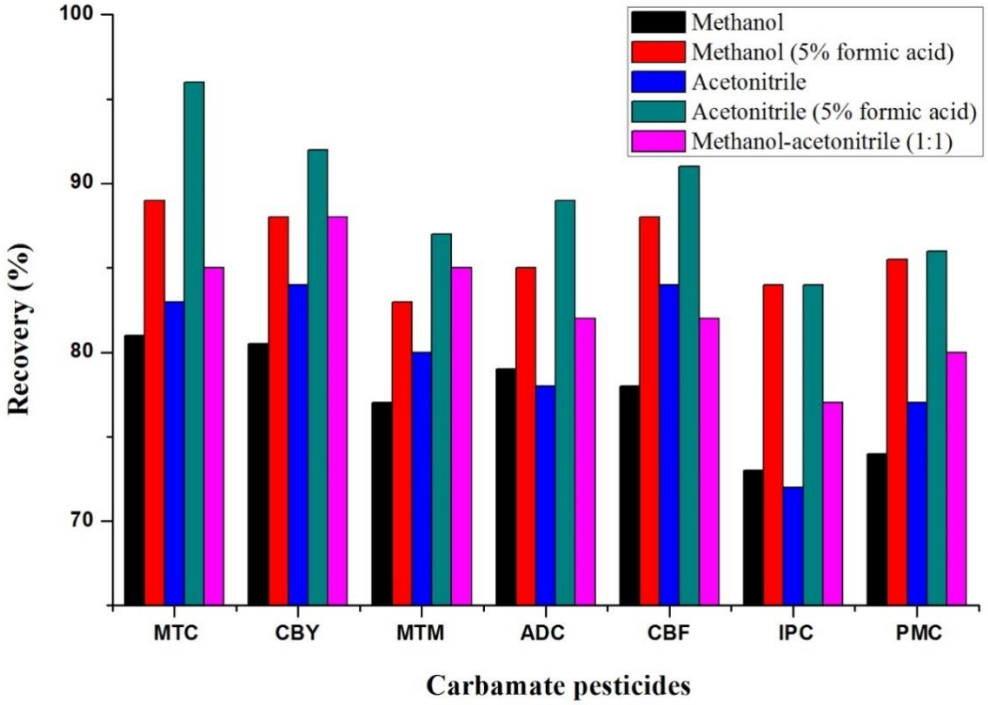


Figure 6. Effects of eluent types for 0.2 g hollow fiber in 10 mL sample solution on the recovery under pH 6, extraction time 35 min and extraction temperature 50℃.

3.2. Choice of capillary electrophoresis parameters

CE conditions affect the separation of target analytes. In this part, the resolution of adjacent effluents was taken as the test index, and the influence of different CE parameters was studied.

3.2.1 pH of separation PBS

The pH of separation PBS used in SPME is an important condition affecting the separation effect. The application range of pH value in the literature work of PBS medium is mostly 5.5-8 [37-42, 44-46]. Therefore, we mainly study the influence of pH in this range on the resolution. When the pH of the PBS changes from 5.5 to 8.0, the resolution of 6 pair of analytes are shown in Figure 7. With the increase of pH value of separation buffer, the resolution of 6 pair of analytes first increased and then decreased, and reached the maximum when pH value was 6.5-7.0. At pH 6.5, the resolution of 5 pair of analytes reached the maximum value, so we chose 6.5 as the pH value of separation buffer solution.

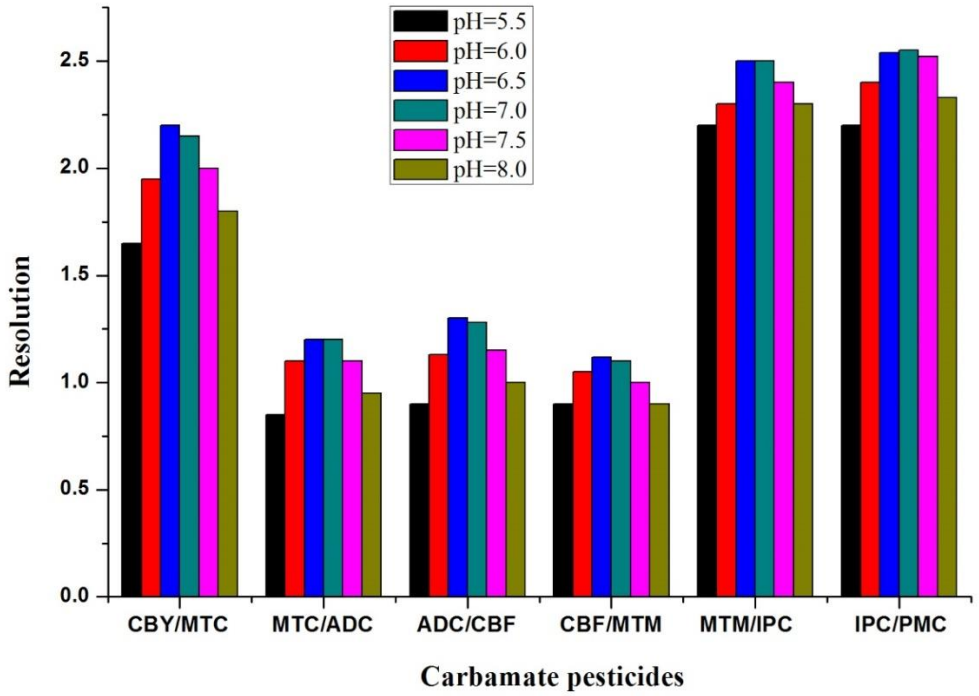


Figure 7. Effects of pH of separation PBS on the resolutions of seven carbamate pesticides at separation voltage 17 kV.

3.2.2 Ionic strength of separation PBS

The ionic strength of separation PBS can be changed conveniently by adding NaCl, Na_2SO_4, KCl or NH_4Cl. The influence of ionic strength on resolution was investigated by adding different concentrations of NaCl in our experiment. As shown in Figure 8, when the concentration of sodium chloride is 45 mmol/L, the resolution of 6 pairs of analytes is relatively large. This conclusion is supported by many literatures [39,41,44-46].

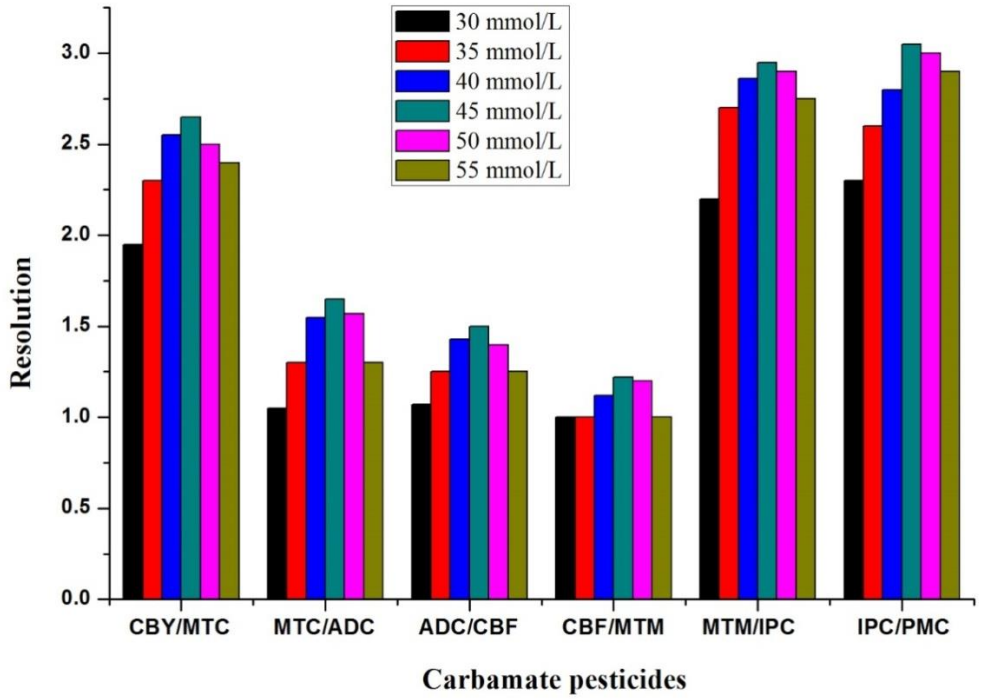


Figure 8. Effects of ionic strength of separation PBS (pH 6.5) on the resolution of 6 pair carbamate pesticides at separation voltage 17 kV.

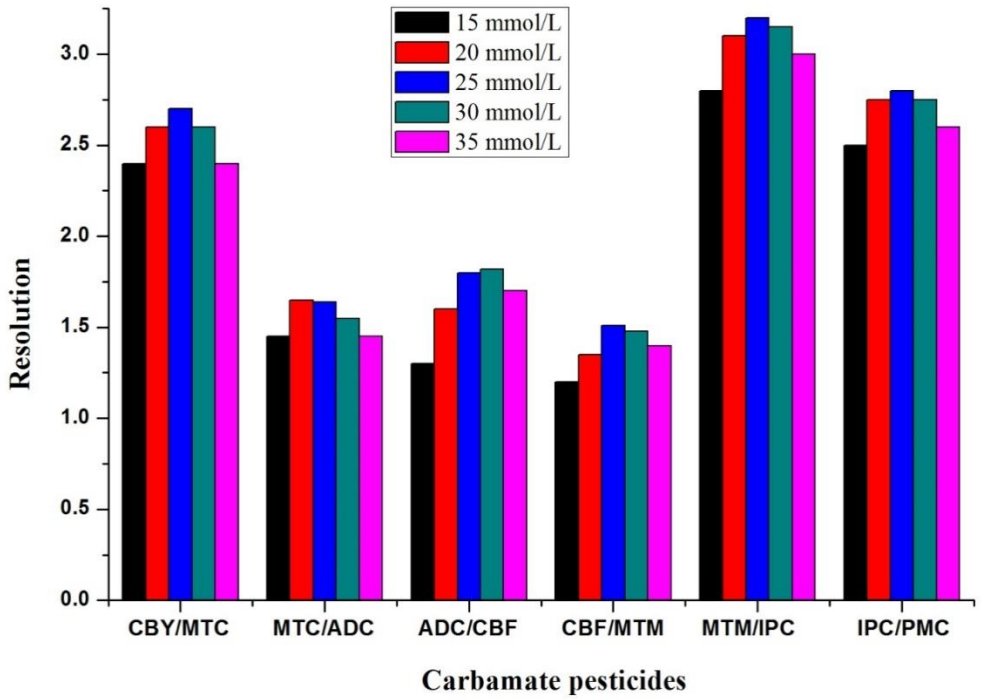


Figure 9. Effects of additive in separation PBS (pH 6.5) containing 45 mmol/L NaCl on the resolution of 6 pair carbamate pesticides at separation voltage 17 kV.

3.2.3 Additive in separation PBS

Cyclodextrin, pyrrolidone, tween 40, isopropanol and other reagents are often added to the separation PBS to improve the separation effect of analytes. Our study found that cyclodextrin has a great influence on the separation of seven carbamate pesticides. The effects of cyclodextrin concentration on the resolution of 6 pair analytes were studied. As shown in Figure 9, when the concentration of cyclodextrin is 25 mmol/L, all of the resolutions of 6 pair analytes are greater than 1.5. The use of additives to improve the separation effect has been confirmed by many experiments [38,40,43,45].

3.2.4 Separation voltage

The separation voltage affects the migration time of components, and then changes the resolution of components. In this experiment, the separation voltages from 15 kV to 19 kV are investigated. The results are shown in Figure 10. Obviously, 17 kV is the best separation voltage. In the literature work, the separation voltage of most experiments is below 20 kV [37-47], which is consistent with our conclusion.

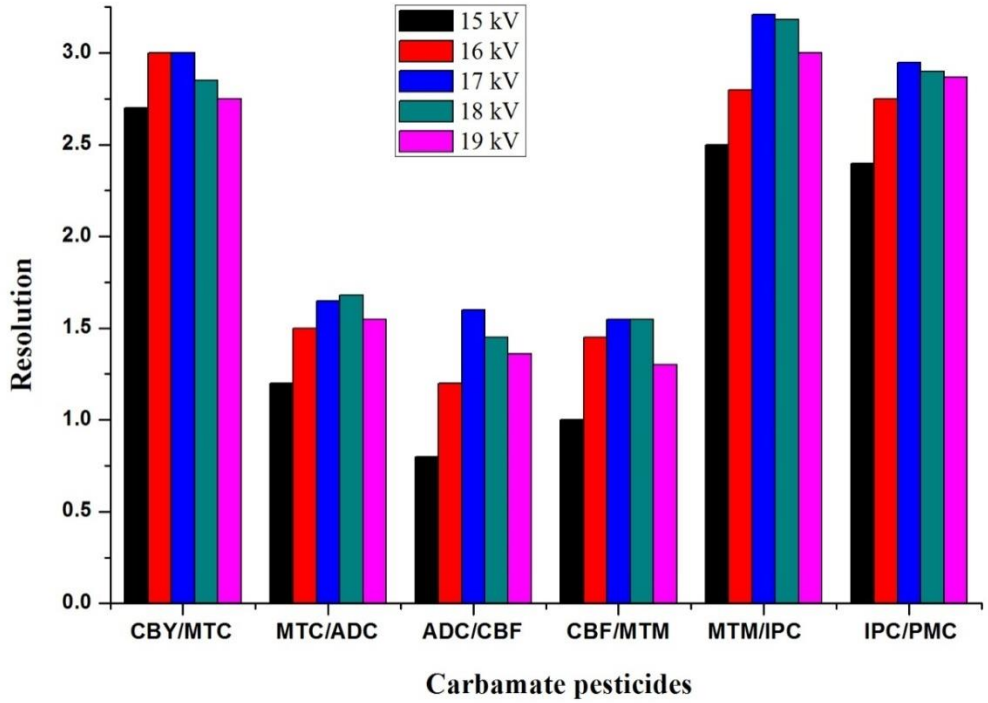


Figure 10. Effects of separation voltage on the resolution of 6 pair carbamate pesticides in separation PBS (pH 6.5) containing 45 mmol/L NaCl and 25 mmol/L cyclodextrin.

3.3 Methodology

The regression equation, linear range and detection limit of this method were investigated by

using carbamate pesticide standard solution. The results were summarized in Table 1. The detection limit of our method is 0.1-0.5 μg/L, which indicates that this method is sensitive.

Table 1. Regression equation, linear range and detection limit of seven carbamate pesticides under the optimized SPME and CE conditions.

Number	Pesticides	Regression equation*	Linear range/(μg/L)	Detection limit/(μg/L)
1	MTC	$I = 110.4C + 24.1$	1.0-1200	0.5
2	CBY	$I = 125.8C + 13.7$	1.0-1200	0.5
3	MTM	$I = 243.6C + 26.1$	0.5-1000	0.2
4	ADC	$I = 284.5C + 34.3$	0.5-800	0.2
5	CBF	$I = 101.2C + 51.2$	1.0-1200	0.5
6	IPC	$I = 78.3C + 15.6$	1.0-900	0.5
7	PMC	$I = 397.7C + 26.0$	0.3-600	0.1

* I: ECL intensity (AU); C: mass concentration, μg/L.

3.4 Sample analysis

The residue and recovery of seven carbamate pesticides in cucumber, tomato and cabbage were studied. The recoveries of seven pesticides in them are 86.1% - 115.8%, which shows that this method is reliable. Residual carbamate pesticides were detected in all three samples, indicating the universality of pesticide residues. The results are shown in Table 2.

Table 2. Analysis results of actual food samples under the optimized SPME and CE conditions.

Pesticides	Measured value (μg/kg)			Added value	Recovery (%, n=7)		
	Cucumber	Tomato	Cabbage	(μg/L)	Cucumber	Tomato	Cabbage
MTC	ND*	ND	3.2	100	89.8	102.5	98.6
CBY	7.3	13.4	ND	100	90.3	114.2	86.1
MTM	ND	0.9	ND	100	88.5	97.1	96.6
ADC	2.4	ND	ND	100	103.2	89.6	102.1
CBF	5.7	4.1	ND	100	105.7	90.4	115.8
IPC	10.1	ND	ND	100	94.8	86.4	97.7
PMC	ND	6.2	3.7	100	87.3	107.4	101.8

*Not detected

4. CONCLUSION

In this paper, a method for simultaneous separation and analysis of seven carbamate pesticides in vegetable samples was established. The method has high sensitivity, wide linear range and good reproducibility, and can be used for rapid determination of carbamate pesticides in vegetable.

ACKNOWLEDGEMENTS
This work was supported by Beijing Natural Science Foundation of China (Grant No.2152013).

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