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# Using nanostructured conductive carbon tape modified with bismuth as the disposable working electrode for stripping analysis in paper-based analytical devices

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ABSTRACT

Low cost disposable working electrodes are specifically desired for practical applications of electrochemical detection considering maturity of electrochemical stations and data collection protocols. In this paper double-sided conductive adhesive carbon tape with nanostructure was applied to fabricate disposable working electrodes. Being supported by indium tin oxide glass, the prepared carbon tape electrodes were coated with bismuth film for stripping analysis of heavy metal ions. By integrating the bismuth modified electrodes with paper-based analytical devices, we were able to differentiate Zn, Cd and Pb ions with the sample volume of around 15 μL. After the optimization of parameters, including modification of bismuth film and the area of the electrodes, etc., Pb ions could be measured in the linear range from 10 to 500 μg/L with the detection limit of 2 μg/L. Our experimental results revealed that the disposable modified electrodes could be used to quantify migrated lead from toys with the results agreed well with that using atomic absorption spectrometry. Although bismuth modification and stripping analysis could be influenced by the low conductivity of the carbon tape, the low cost disposable carbon tape electrodes take the advantages of large-scaled produced double-sided carbon tape, including its reproducible nanostructure and scaled-up fabrication process. In addition, the preparation of disposable electrodes avoids time-consuming pretreatment and experienced operation. This study implied that the carbon tape might be an alternative candidate for practical applications of electrochemical detection.


## 1. Introduction

In electrochemical detection, the working electrode plays a pivotal role because all the electrochemical reactions occur on its surface. There have been numerous studies focusing on the working electrode surface, such as chemical modification electrodes [1,2]. Because of the electrochemical reactions, the surface of working electrodes is delicate and could be easily polluted during electrochemical detection. Such contamination would inevitably make the electrochemical detection unstable and irreproducible. To address this problem, working electrodes need to be frequently refreshed in order to keep a clean and unpolluted surface. Toward this end, the contaminated surface of working electrodes normally should be physically removed for reproducible electrochemical detection results. This process is time-consuming and requires experienced operation so that practical applications of electrochemical detection have been largely limited. Therefore, it is of importance to develop low cost working electrodes for one-time use [3–5].

During the past years, there have been a number of studies on disposable working electrodes for a variety of purposes [6–9]. The most successful disposable working electrodes were screen-printed electrodes because of their simple fabrication and changeable formats [10,11]. Meanwhile, carbon, gold and other nanomaterials have been used for preparation of disposable electrodes with various routes [6,12–14]. For example, disposable carbon electrodes have been reported for liquid chromatographic determination

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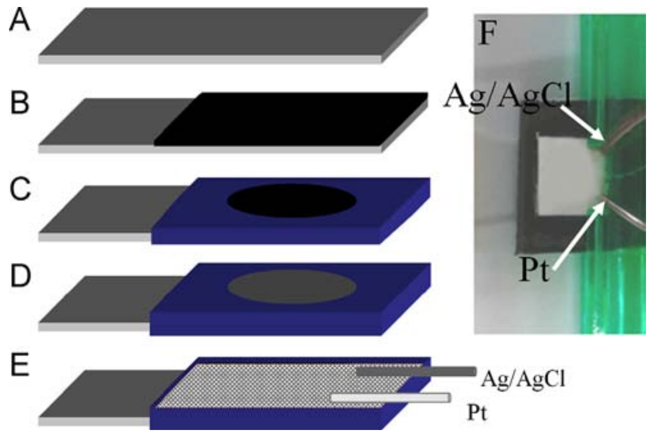



**Fig. 1.** The schematic of the bismuth modified carbon tape electrode and its integration with PAD for Pb detection. (A) ITO glass; (B) a piece of carbon tape was attached on the ITO glass; (C)the insulate plastic adhesive tape with a hole (diameter: 4 mm) was attached on the carbon tape; (D) the bismuth film was electrodeposited on the exposed part of the carbon tape to form the bismuth modified carbon tape electrode; (E) a piece of filter paper with 6 mm long and 6 mm wide, an Ag/AgCl wire and a Pt wire were integrated with the bismuth modified carbon tape to form the PAD for electrochemical detection and (F) the photo of the PAD.

of catecholamines in blood plasma samples based on sputtering carbon on a polyether–etherketone (PEEK) film [15]. Indium tin oxide (ITO) glass was also used as the substrate for the modification of glucose oxidase on gold nanoparticles for rapid glucose measurement [16]. It should be emphasized that the price of ITO glass has significantly decreased with the development of modern display technology. However, currently most of disposable electrodes still suffer from expensive devices, time-consuming pretreatment or unsatisfactory reproducibility. It is believed that engineered materials would be more preferable for the fabrication of disposable electrodes.

Stripping analysis has been extensively investigated for the detection of trace heavy metal ions and biochemical analysis [17,18]. Initially, mercury (such as hanging mercury electrodes) was used as the material of working electrodes since it could be refreshed conveniently for reproducible detection [19,20]. However, the strong toxicity of mercury makes its applications unfriendly to the environment. To address this problem, researchers have pursued alternative materials, such as bismuth and other materials, etc., to modify electrodes for stripping voltammetry [5,21–26]. The advantages of bismuth film include low toxicity, wide potential range, and insensitivity to dissolved oxygen. On the other hand, various materials, including graphite [27], pencil [8], carbon paste [28,29] and gold [30,31], have been used as the substrate of working electrodes in stripping analysis, which is more active than in other electrochemical analytical approaches because of its well-know mechanisms. The development of low-cost disposable working electrodes could considerably shrink the gap between fundamental research and practical applications of stripping analysis and other electrochemical detections.

In our previous report, double-sided conductive adhesive carbon tape supporting on ITO glass was used as the substrate of working electrodes in order to stabilize electrochemiluminescence emission from quantum dots [32]. In this paper, we regulated the areas of the carbon tape electrodes and then modified them with bismuth for stripping analysis. Our experimental results showed that the bismuth modified carbon tape electrodes could be used to effectively differentiate Zn, Cd and Pb ions in the paper-based analytical devices. Specifically, the disposable working electrodes could be used to quantify the concentration of Pb ions migrated from toys with similar results obtained using atomic absorption spectrometry (AAS). Our investigation implied that carbon tape might be a potential candidate for the fabrication of low cost disposable electrodes.

## 2. Materials and methods

### 2.1. Chemicals and materials

All the chemicals were of analytical grade unless otherwise mentioned. $Pb(NO_3)_2$ and $Bi(NO_3)_3 \cdot 5H_2O$ were from Runjie Chemical Reagent Company (Shanghai, China). Sodium acetate anhydrous ($CH_3COONa$) was from Xilong Chemical Co., Ltd (Guangzhou, China). Acetic acid was from Shenbo Chemical Co., Ltd (Shanghai, China). The double distilled water was collected from SZ-93A automatic double water distiller made by Shanghai Yarong Biochemical Instruments (Shanghai, China). The stock solution of Pb(II) (1000 $\mu g\, L^{-1}$) was prepared using acetate buffer solution (0.1 M, pH 4.5) as the supporting buffer and diluted to be certain concentrations. The qualitative filter papers (Whatman No. 1) were from Whatman International Ltd. (Maidstone, United Kingdom). The Indium tin oxide (ITO) conductive glass ($355.6 \times 406.4 \times 1.1$ mm STN, 10 Ω) was purchased from Nanbo Display Technology Co. LTD (Shenzhen, China). The conductive double-sided carbon adhesive tape (12 mm wide, 0.16 mm thick and 20 m long) was purchased from SPI Supplies (West Chester, PA, USA). Toy samples consisting of Pb were obtained from Chinese Academy of Inspection and Quarantine.

### 2.2. Sample preparation

The toys were treated according to the national safety technical regulation for toys of China (GB 6675-2003). Briefly, they were cut or crushed at room temperature and then mixed with 0.07 M HCl at $37 \pm 2$ °C. Then the mixture was shaken for 1 min. After that, 2 M HCl was added to adjust the pH value to be 1.0–1.5. The mixture was stirred for 1 h and kept for 1 h at $37 \pm 2$ °C without light. Then the mixture was filtrated with a membrane filter and the sample solutions were mixed with acetate buffer solution (0.1 M, pH 4.5) for detection with stripping voltammetry and AAS, respectively.

### 2.3. The bismuth modified carbon tape electrode

As shown in Fig. 1, ITO glass was cut to be 20 mm long and 7 mm wide. Then the conductive carbon tape (12 mm long and 7 mm wide) was attached on the conductive surface of ITO glass. A section of plastic adhesive tape was punched with a hole (4 mm diameter) and then attached on the carbon tape in order to provide an identical area for detection. The carbon tape electrode was then electrodeposited with bismuth using 60 μl of 60 $mg\, L^{-1}$ bismuth nitrate in 1.5 $molL^{-1}$ HCl solution containing 0.15 $molL^{-1}$ sodium citrate by applying the potential of −1.8 V for 120 s [33]. We found that the selection of the buffer solution is critical in order to fully solve bismuth. After that, the bismuth modified carbon tape electrode was rinsed with double distilled water and dried for use.

### 2.4. Stripping voltammetry and other analysis

Stripping voltammetry was carried out on a CHI1230B electrochemical working station (CH Instrumentation, Shanghai, China) with a three-electrode system using the modified carbon tape electrode as the working electrode, an Ag/AgCl wire as the reference electrode and a platinum wire as the counter electrode. A piece of the Whatman filter paper No. 1 with 6 mm long and 6 mm wide was put above the surface of the bismuth modified carbon tape electrode. Sample solutions in 0.1 M acetate buffer (pH 4.5) with the volume of 15 μL was dropped on the filter paper for stripping voltammetric analysis. The square-wave anodic stripping voltammetry (SWASV) was performed with frequency at 20 Hz, potential step at 5 mV, and amplitude at 25 mV. The procedure consisted of preconcentration at −1.5 V for 120 s, equilibration period for 60 s, and squarewave voltammetric stripping scan from −1.5 to 0.1 V. The preconcentration time was 120 s for the samples consisting of Zn, Cd and Pb ions. For the determination of Pb ions, the preconcentration time was 300 s. Between every experiment, the counter electrode and reference electrode were washed thoroughly with double distilled water. The concentration of Pb ions in the sample solutions prepared from toys was simultaneously measured with atomic absorption spectrophotometer 360MC (The Third Analytical Instrument Factory, Shanghai, China).

## 3. Results and discussion

### 3.1. Design of the low cost working electrode

For disposable working electrodes, it is preferred that the materials are low cost and their fabrication steps are simple. Herein double-sided conductive adhesive carbon tape was tested as the material to fabricate the disposable working electrodes. The SEM image in Fig. 2 illustrated the nanostructure of the carbon tape at the level of ca. 100 nm. It is believed that such nanostructure of the carbon tape could help the generation of stable and reproducible electrochemiluminescent emission [32]. We assumed that carbon tape with such structure could enhance other electrochemical detections. In addition, the fresh surface of carbon tape could be used to fabricate disposable electrodes because its two sides are protected well before use. Thus, the disposable electrodes could be fabricated without need of complicated devices and time-consuming pretreatment. Moreover, one unique advantage of the carbon tape electrodes is their low cost. In our approach, the carbon tape with the area of 12 mm long and 7 mm wide was applied for one working electrode. Thus, a roll of carbon tape with the length of 20 m and the price of \$43 could be used to fabricate about 2800 electrodes. Indium tin oxide (ITO) glass was used as the support in order to enhance the conductivity of carbon tape for electrochemical detection. With the advance of modern technology, the price of ITO glass has been significantly decreased. For the area of 35.5 cm wide and 40.6 cm long, the price of ITO glass is around \$35. ITO glass with such a price could be applied for around 1000 electrodes. As a result, for one disposable working electrode made with the carbon tape and ITO glass the price would be about \$0.05 for the materials. Such a low price and the convenient fabrication process make the carbon tape electrode attractive for one-time use.

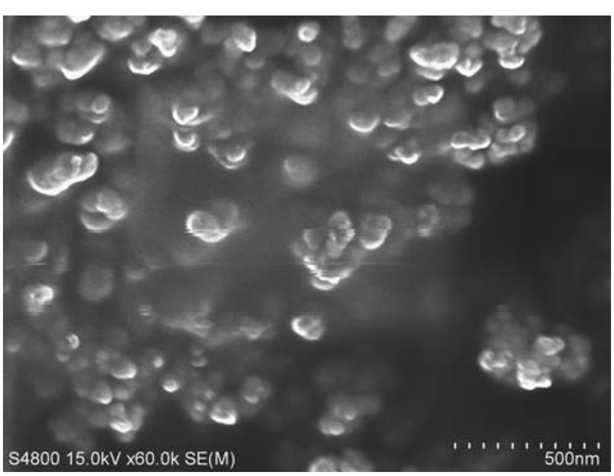



**Fig. 2.** The SEM image of double-sided conductive adhesive carbon tape.

### 3.2. Optimization of stripping analysis

The carbon tape electrodes were then modified with bismuth film for stripping analysis. For this purpose, the solution of bismuth could be prepared with acetate solution (pH 4.5) or 1.5 $molL^{-1}$ HCl containing 0.15 $molL^{-1}$ sodium citrate [33–35]. However, in acetate solution bismuth could not be fully solved. Although the mixture of bismuth nitrite and acetate solution has been commonly applied on other carbon-based electrodes [34,35], the carbon tape could not be reproducibly modified with stable bismuth film. By comparison, the stable bismuth film could be obtained on the carbon tape electrodes using the solution of

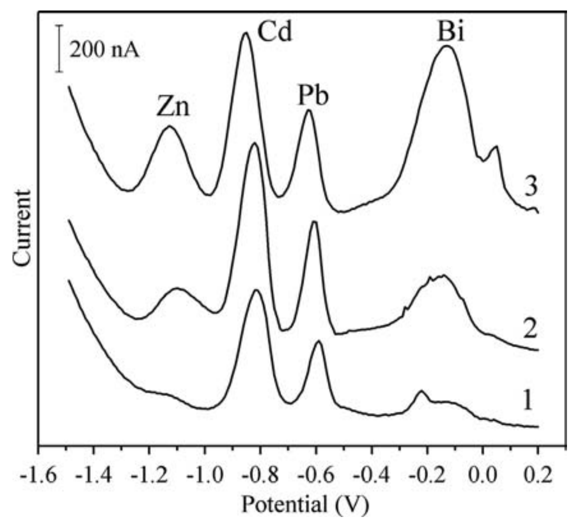



**Fig. 3.** Stripping analysis of 100 μg $L^{-1}$ Zn, Cd and Pb ions with the bismuth modified carbon tape electrode prepared with the bismuth concentration of (1) 20 mM, (2) 60 mM and (3) 100 mM. Experimental parameters: frequency, 20 Hz; a potential step, 5 mV; amplitude, 25 mV; equilibration time, 60 s; preconcentration time, 120 s; deposition voltage for bismuth film, −1.8 V; deposition time for bismuth film, 120 s.

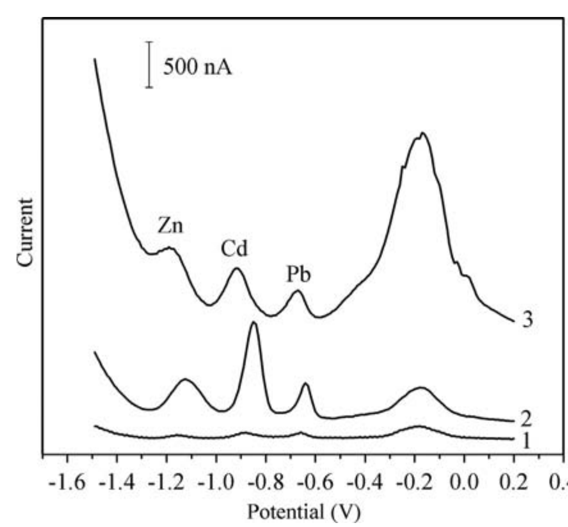



**Fig. 4.** Stripping analysis of 100 μg $L^{-1}$ Zn, Cd and Pb ions using the bismuth modified carbon tape electrodes with the area of (1) A circle with the diameter of 2.5 mm; (2) a circle with the diameter of 4 mm and (3) a rectangle with 7 mm long and 6 mm wide. Experimental parameters: the same as in Fig. 3.

bismuth in HCl consisting of sodium citrate. The differences between the carbon tape electrodes and other carbon-based electrodes might be attributed to the fact that the conductivity of the carbon tape is not as good as other carbon materials. Such differences revealed that the solution of bismuth is critical for the modification of bismuth on the carbon tape electrode. The solution of bismuth in 1.5 $molL^{-1}$ HCl containing 0.15 $molL^{-1}$ sodium citrate was chosen in our following experiments.

Fig. 3 illustrates the influence of bismuth concentration on stripping analysis of three heavy metal ions (Zn, Cd and Pb). The peak heights of Zn, Cd and Pb ions increased from 15, 497 and 279 to 122, 778 and 478 nA, respectively, with the increase of bismuth concentration from 20 mM to 60 mM. With the further increase of bismuth concentration to100 mM, the peak heights of Cd and Pb ions decreased to 706 and 408 nA, respectively, while that of Zn ions increased to 283 nA. In addition, it could be observed that all the peak potentials slightly shifted to the negative side with the increase of bismuth concentration. All the peak potentials of three metal ions were similar to those reported using the bismuth film modified electrodes [36]. In addition, the peak height of Bi gradually increased with the increase of bismuth concentration from 20 to 100 mM. Considering the peak heights, the bismuth concentration of 60 mM was chosen in our following investigation.

Fig. 4 shows the influence of the working electrode area on stripping analysis. It could be observed that with the decrease of the area from 42 $mm^2$ (a rectangle with 7 mm long and 6 mm wide) to ca. 12.6 $mm^2$ (a circle with the diameter of 4 mm), the peak heights for the Zn, Cd and Pb ions increased from 304, 465 and 294 nA to be 338, 990 and 360 nA, respectively, with all the peaks becoming sharper. With the further decrease of the area to be ca. 4.9 $mm^2$ (a circle with the diameter of 2.5 mm), all the peak heights decreased dramatically. Thus, the area of ca. 12.6 $mm^2$ was applied for the determination of heavy metal ions based on stripping analysis.

Fig. 5 shows the dependence of peak heights of three metal ions on the concentrations in stripping analysis using the bismuth modified carbon tape electrodes. It could be observed that with the increase of the concentration from 100 to 200 $\mu g\ L^{-1}$, peak heights increased from 338, 987 and 359 nA to 521, 1358 and 569 nA for Zn, Cd and Pb ions, respectively. Such results implied that the bismuth modified carbon tape electrodes could potentially be used to quantify the concentration of multiple metal ions.

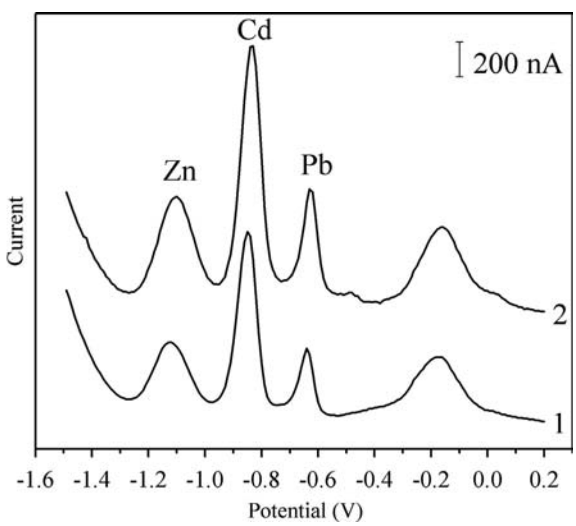



**Fig. 5.** Stripping analysis of (1) 100 $\mu g\ L^{-1}$ and (2) 200 $\mu g\ L^{-1}$ Zn, Cd and Pb ions with the prepared bismuth modified carbon tape electrode. Experimental parameters: the same as in Fig. 3.

### 3.3. Determination of Pb migrated from toy samples

In the following we focused on the determination of Pb ions with the bismuth modified carbon tape electrodes. It is worth to note that the preconcentration time was optimized to be 300 s for the quantification of Pb ions. As shown in Fig. 6, with the increase of $Pb^{2+}$ concentration the peak height increased accordingly. Interestingly, the peak height and the concentration could be correlated with two sections of linear curves. Within the concentration from 0 to 80 $\mu g\ L^{-1}$ the curve could be represented with a line passing with the slope of 1.26 $nA \cdot \mu g^{-1}$ L (correlation coefficient, 0.98). For the concentration in the range from 80 ppb to 500 ppb the relationship could be fitted with a line consisting of a slope of 3.16 $nA \cdot \mu g^{-1}$ L and in intercept of −138 nA (correlation coefficient, 0.96). Their different fitting results might be attributed to the increased conductivity due to the increase of Pb preconcentrated on the modified electrodes. The detection limit of Pb ions was estimated to be ca. 2 $\mu g\ L^{-1}$ based on the signal to noise ratio of 3. The linear range and detection limit of $Pb^{2+}$ concentration were similar to those reported using stripping analysis [37].

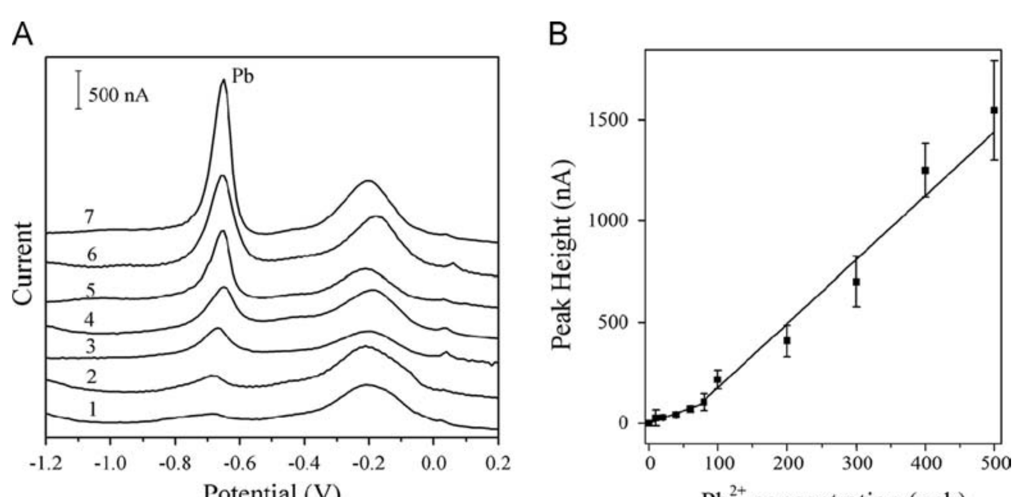
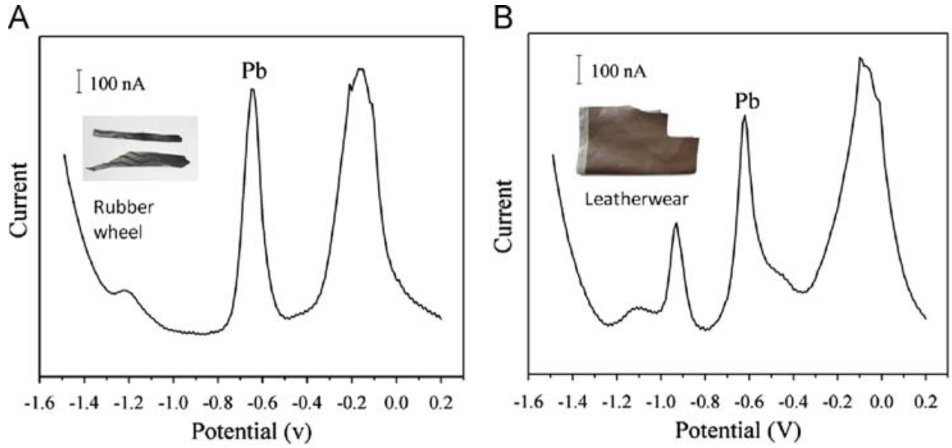
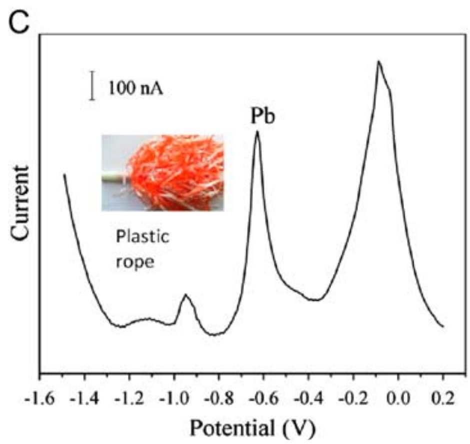



**Fig. 6.** The dependence of peak current on the concentration of Pb ions (A) and the linear fitting (B) for the relationship between the peak height and $Pb^{2+}$ concentration. Experimental parameters: preconcentration time, 300 s; the others, the same as in Fig. 3.

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| Sample | Detection result using the atomic absorption spectrometry (ppb) | Detection results with the bismuth modified carbon tape electrode (ppb) |
|---|---|---|
| Rubber wheel | 425.2 | 437.5 ± 39.4 |
| Leather wear | 375.6 | 412.3 ± 70.1 |
| Plastic rope | 300.0 | 299.2 ± 35.9 |

**Fig. 7.** Stripping analysis of Pb ions migrated from (A) the rubber wheel of a child bicycle, (B) leatherwear and (C) plastic rope. Experimental parameters: preconcentration time, 300 s; the others, the same as in Fig. 3.

It needs to be emphasized that all the bismuth carbon tape electrodes were used for just one time and every data point was obtained using five different carbon tape electrodes. Such results revealed that the bismuth modified carbon tape electrodes could be practically applied for determination of Pb ions.

Finally, the bismuth modified carbon tape electrodes were applied to measure Pb ions migrated from three toys samples (a rubber wheel from a child bicycle, a section of leatherwear and a plastic rope) under the acid condition similar to gastric juice. As shown in Fig. 7, for all three toys there was a well-defined sharp peak at ca −620 mV, which could prove the existence of Pb ions. The detection results with stripping voltammetry agreed well with those obtained from AAS. Interestingly, the peaks at the potential of around −0.95 V might imply the existence of Cd migrated from the toys (the leatherwear and the plastic rope). Such results revealed the potential of the double-sided conductive adhesive carbon tape in practical applications of electrochemical detection.

## 4. Conclusion

In summary, herein the double-sided conductive adhesive carbon tape was used as the substrate to fabricate low cost disposable working electrodes. The preparation of carbon tape electrodes with the cost of $0.05 per electrode avoided expensive instruments, experienced operation and time consuming pretreatment. The carbon tape electrodes are superior because of nano-structured surface and large-scale engineered fabrication process of the carbon tape. In addition, the bismuth modified carbon tape electrodes could be integrated with the paper-based analytical devices for effective stripping analysis of Pb and other metal ions with the sample volume of only 15 μL. Those characteristics of the carbon tape electrodes make their practical applications attractive for electrochemical detection in environmental monitoring and other in situ areas.

## Acknowledgments

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