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REVIEW

RECENT RESULTS IN FOOD ANALYSIS WITH CARBON PASTE ELECTRODES: ORGANIC CONSTITUENTS, ADDITIVES AND CONTAMINANTS

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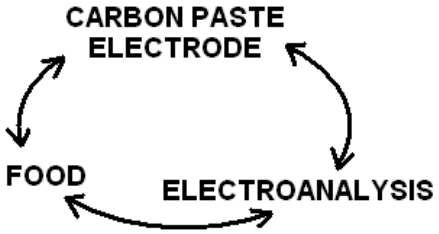
This paper reviews the electroanalytical determination of organic compounds in foods using carbon paste electrodes. Thirty-six original research papers were analyzed from the last five year period (January, 2009 − October, 2013) to give insight in the role of carbon paste electrodes in the determination of organic constituents, additives and contaminants in different foodstuffs, except drinking water. The present paper includes also a brief overview of food analysis, with special emphasis on electroanalysis and carbon paste electrodes therein.



INTRODUCTION

Nowadays, consumers are taking unprecedented interest in the way food is produced, processed and marketed, and are increasingly calling for their Governments to accept greater responsibility for food safety and consumer protection.[1]

To guarantee food safety and quality and satisfy the requirements of the consumer, it is necessary to ensure that efficient analytical methodologies are possessed by the food industry.[2] The classical analytical methods used at the beginning of the 20th century based on the so-called "wet chemistry" have evolved into the powerful instrumental techniques used in food laboratories. This improvement has led to significant enhancements in analytical accuracy, precision, detection limits, and sample throughput, thereby expanding the range of practical applications in food analysis.[3]

Traditionally, analytical techniques have been classified according to their working principle. Every technique provides specific information on the sample or components under study based on a specific physical-chemical interaction, and all have their own advantages and drawbacks when applied to food analysis.[3]

With the appearance of advanced approaches such as screenprinting technology, biosensors, microchips and nanotechnology, among others, electroanalysis is undergoing a true Renaissance and offers a high potential in the field of food analysis, especially in common labs where sophisticated analytical instrumentation is not available. There are more arguments for application of electroanalysis in the analytical chemistry of foods. Firstly, many analytes in foods are electroactive, and electrochemical detection offers good selectivity (especially after electrode

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modification with different (bio)(nano)materials) and sensitivity for their direct detection without the need of the derivatization step. On the other hand, electroanalytical instrumentation can be easily miniaturized without loss of analytical performance, has a relatively low cost, and can be used for the investigation of samples with low turbidity. Furthermore, electrochemical sensing can operate in batch and in continuous regimes, coupled as detectors in advanced separation techniques and as a transduction system in biosensors.[4]

Among the electrodes of choice in the electroanalytical chemistry, the so-called carbon paste electrodes[5-9] (CPEs) offer a number of advantageous features: broad potential window (mainly at the anodic side), very low residual currents (background), unique surface characteristics, low cost, simple preparation (directly in labs), usually low toxicity, and almost countless possibilities of their chemical and biological modifications.[7-11] The bare (binary, unmodified) CPE is a mixture of graphite (carbon) powder and suitable (liquid) binder, which is packed into an appropriate electrode body.[12] Both main components, as well as their mutual ratio then co-determine the physicochemical and electrochemical properties that can further be changed by adding a third constituent (modifier, catalyst, mediator, stabilizer, etc.). In these cases, one obtains chemically or biologically modified CPEs[10] and CP-biosensors,[13] respectively. The bare carbon paste mixtures are being made mostly from highly pure spectroscopic graphites and mineral or silicone oils (and greases). Whereas these mixtures are called traditional carbon pastes,[8,11] there is also a wide spectrum of other types being classified as special carbon pastes.

The literature about CPEs is quite broad and includes thousands of original research articles and many reviews. The very first review on CPEs had been published by the inventor – Ralph N. Adams – himself.[12] Another related elaborate came a quarter of a century later; being focused predominantly on chemically modified CPEs[10] and soon followed by two similar works.[11,14] In the recent years, the widespread area of CPEs has been reviewed in a series of exclusive articles,[6,15-18] in a chapter in the Encyclopedia of Sensors[8] and also in form of a detailed monograph.[7] The possibilities and limitations of CPEs in the determination of organic compounds were exclusively reviewed recently.[19,20] An extensive retrospective review article was published in 2009[17] about the applicability of CPEs in food analysis.

The recent review will collect and analyze the last achievements in the application of CPEs in the determination of organic compounds in foods in the last five year period (2009−2013).

PRESENT RESULTS

Investigated organic analytes

Table 1 shows organic analytes that have been determined with CPEs (including CPE biosensors also) in different foodstuffs in the period of 2009−2013. The applied electroanalytical method/ technique (with selected performance characteristics), the composition of the working electrode, the investigated food sample and the applied sample preparation procedures are in all cases referred together with the target analyte.

The most important organic food constituents determined with CPEs in the last years were: carbohydrates (glucose, fructose),[21,22] vitamins (ascorbic acid, folic acid),[23-28] natural (poly)phenols (catechol, morin, gallic acid or total polyphenols as such)[29-32] and adenosine triphosphate (ATP).[33] Aroma compounds, like vanillin[34] were also investigated. Xanthine[35] and biogenic amines[36] (cadaverin and putrescine) − as important meat freshness indicators − were successfully determined in different fish products. Among food additives, the mostly analyzed group was undoubtedly the group of food colorants (brilliant blue, tartrazine, sunset yellow, sudan I, ponceau 4R).[37-44] Concerning food contaminants analyzed with CPEs, pesticides (cyanazine, propazine, paraquat, primicarb, carbofuran, carbaryl, formetanate, ziram)[45-50] were the most numerous. Biotoxins (zearalenone, α-zearalenol, β-zearalenol, brevetoxin B, citrinin),[51-53] pharmaceutical residues (sulfamethoxazole and different tetracycline-type antibiotics)[54,55] and different xenoestrogens (pentachlorophenol, bisphenol-A, 2,4-dichlorophenol, 4-tert-octylphenol, 4-nonylphenol)[56] were successfully determined as well.

Samples

The list of the food samples investigated with CPEs is very broad and diverse. The mostly investigated samples are undoubtedly from the group of the drinks (beverages), first of all fruit and vegetable juices, soft and energy drinks, and wines.[21-28,32,37-41,44] Most of them were analyzed without special sample preparation (except dilution

in some cases). The more complex samples (different raw meat and meat products, eggs, tea, fruit, vegetable and honey samples, olives and olive oil, rice, flour, milk powder, different spices, sauces, jellies, candies, chocolate)[22,29-33,35,36,39-43,45-56] – with a few exceptions − were investigated after different sample pretreatment procedures like dilution, homogenization, squeezing/grinding/ chopping, dissolution in acid, various extraction procedures, filtering, etc.

Table 1

Determination of organic compounds in foods using CPEs (analytes, techniques/methods, working electrode constituents, investigated food samples, sample preparation procedures, selected performance characteristics, references)

Analyte	Technique / method	Working electrode (basic constituents of the CPE if available)	Food sample	Sample preparation (selected parameters if available)	Linear range (LOD, acc. time / resp. time)	Ref.
glucose	amperom.	NiO nanoparticles-modified CPE (graphite powder+paraffin)	juice	dilution	1-110 μM (0.16 μM, ---)	21
fructose	amperom.	fructose dehydrogenase and osmium redox polymer (poly(1-vinylimidazole)$_{12}$-[osmium(4,4´-dimethyl-2,2´-dipyridyl)$_2$Cl$_2$]$^{2+/+}$)-modified CPE (SWCNT+mineral oil)	honey, fruit juices, soft and energy drinks	dilution	0.1-5 mM (1 μM, ---)	22
ascorbic acid	DPV	MWCNT/THAI (tetraheptylammonium iodide)/I_2-modified CPE (graphite powder+paraffin oil)	orange juice	---	0.056-12 mM (0.036 mM, ---)	23
ascorbic acid	DPV	MWCNT/CTAI (cetyltrimethylammonium iodide)/I_2-modified CPE (graphite powder+paraffin oil)	orange juice	---	0.056-12 mM (0.0012 mM, ---)	24
ascorbic acid	DPV	NiO nanoparticles-modified CPE (graphite powder+paraffin oil)	fruit juices / vegetable juices	mechanical squeezing and filtration / grating and centrifugation	0.1-700 μM (0.05 μM, ---)	25
ascorbic acid	DPV	*p*-aminophenol and MWCNT-modified CPE (graphite powder+paraffin oil)	fruit juices / vegetable juices	mechanical squeezing and filtration, acidification / grating and centrifugation	0.2-120 μM (0.08 μM, ---)	26
ascorbic acid	DPV	ZnO/CNT-modified CPE (graphite powder+paraffin oil+ ionic liqid (1-methyl-3-butylimidazolium bromide))	fruit juices / vegetable juices	mechanical squeezing and filtration; acidification / grating and centrifugation	0.1-450 μM (0.07 μM, ---)	27
folic acid	SWV	ZnO nanoparticles-modified CPE (graphite powder+paraffin oil+ ionic liqid (1-butyl-3-methylimidazolium hexafluorophosphate))	apple juice	centrifugation; filtering; dilution	0.05-1.5 and 1.5-550 μM (0.01 μM, ---)	28
catechol	DPV	Al/SiO_2-modified CPE (graphite powder+paraffin oil)	tea	extraction with 20% (v/v) methanol; filtration	0.5-50 μM (0.1 μM, ---)	29
morin	amperom. (detector in SI-LOV)	polyvinylpyrrolidone-modified CPE (graphite powder+paraffin oil)	tea	extraction with ether/acetone (14:1, v/v); centrifugation	1-10 and 10-400 μM (0.19 μM, ---)	30
gallic acid	DPV	TiO_2-modified CPE (graphite powder+paraffin oil (nujol))	black and green tea	dissolving in nitric acid; heating to dryness; dissolving in water; filtration	2.5-150 μM (0.94 μM, ---)	31
total polyphenols (as t-resveratrol (1) and caffeic acid (2))	amperom.	ferrocene and peroxidase (from Brassica napus hairy roots)-modified CPE (MWCNT+mineral oil)	red and white wines / black, red and green teas	--- / extraction with hot water; filtration	1: 0.219-228 μM (0.101 μM, ---) 2: 0.333-383 μM (0.111 μM)	32

Table 1 (continued)

ATP	SW AdSV (detection system in a double-surface competitive assay)	Au nanoparticles and graphene-modified CPE (graphite powder+mineral oil)	pericarp tissue of banana and litchi, tomato seed	frozing; powdering; ATP extraction using different procedures for fruits and vegetable	0.114 nM-3.42 μM (20.1 pM, 12 min)	33
vanillin	DPV	ionic liqid (1-butyl-3-methylimidazolium tetrafluoroborate)-modified CPE (---)	---	---	13.1-197 μM (6.57 μM, ---)	34
xanthine	amperom.	CPE surface modified with CNFs (graphite powder+mineral oil)	fish meat	homogenization in 10% HCl; dilution with double distilled water; centrifugation; filtration	0.03-21.19 μM (0.02 μM, ---)	35
biogenic amines (cadaverin (1), putrescine (2))	DPV	MnO_2-modified CPE surface modified with pea seedling amine oxidase (PSAO)-containing nafion film (carbon powder+paraffin oil)	fish sauce	---	1: 0.294-0.861 mM (97.8 μM, ---) 2: 0.272-0.76 mM (90.7 μM, ---)	36
brilliant blue (1), tartrazine (2)	DPV	MWCNT-modified CPE (graphite powder+paraffin oil)	soft drinks	dilution	1: 0.05-22 μM (9 nM, ---) 2: 0.05-25 μM (5 nM, ---)	37
tartrazine (1), sunset yellow (2)	DP AdASV	Au nanoparticles-modified CPE (graphite powder+paraffin oil)	soft drinks	---	1: 0.05-1.6 μM (2 nM, 60 s) 2: 0.1-2 μM (30 nM, 60 s)	38
tartrazine	potentiom.	ion exchanger (reaction product of tartrazine and cetryltrimethyl ammonium bromide)-modified CPE (graphite+dioctyl phthalate)	soft juice / solid fruit (strawberry) jelly and custard powder	filtration / homogenization; dissolving in hot water; filtration	0.83 μM-10 mM (0.47 μM, ---)	39
tartrazine	potentiom.	ion exchanger (reaction product of tartrazine and cetryltrimethyl ammonium bromide)-modified CPE (graphite+tris(2-ethylhexyl) phosphate)	soft juice / solid fruit (strawberry) jelly and custard powder	filtration / homogenization; dissolving in hot water; filtration	0.43 μM-10 mM (0.32 μM, 5-8 s)	40
tartrazine (1), sunset yellow (2)	SWV AdASV	graphene and TiO_2-modified CPE (graphite+paraffin oil)	candy, royal jelly, ice cream, solid custard jelly, juice powder/ soft drink / colouring coated chocolate	dissolving in hot water; dilution; filtration / --- / coloured shell dissolving in distilled water; solid parts separation; centrifugation; dilution; dye extraction with wool yarn; dye recovery from yarn	1: 0.02-1.18 μM (8 nM, 2 min) 2: 0.02-2.05 μM (6 nM, 2 min)	41
sudan I	DP AdASV	unm. CPE (expanded graphite+solid paraffin)	chilli sauce, ketchup	extraction with ethanol; filtration	5 nM-7 μM (0.9 nM, 300 s)	42
sudan I	SWV	Pt/MWCNT and ionic liquid (1-methyl-3-butylimidazolium bromide)-modified CPE (graphite+paraffin oil)	chilli, tomato and strawberry sauce, chilli powder	extraction with ethanol; filtration	8 nM-600 μM (3 nM)	43
ponceau 4R	---	Ca-montmorillonite modified CPE (---)	soft drinks	---	16.56 nM-4.97mM (4.14 nM, ---)	44

Table 1 (continued)

cyanazine	DP AdCSV	molecularly imprinted polymer-modified CPE (graphite powder+paraffin oil)	vegetables / rice	juicing and filtration / drying; powdering; suspending in solvent; filtration	5-1000 nM (3.2 nM, 200 s)	45
cyanazine (1), propazine (2)	DP AdCSV	molecularly imprinted polymer-modified CPE (graphite powder+*n*-eicosane)	vegetables / rice	juicing and filtration / drying; powdering; suspending in solvent; filtration	1: 0.05-9 μM (0.01μM, 200 s) 2: 0.01-1 μM (0.001μM, 200 s)	46
propazine	DP AdCSV	molecularly imprinted polymer-modified CPE (graphite powder+paraffin oil)	vegetables / rice	juicing; centrifugation / drying; powdering; suspending in solvent; filtration	0.01-1 and 1-55 μM (1 nM, 3 min)	47
paraquat	SW AdASV	chitin-modified CPE (---)	olives, olive oil	homogenization (olives); water extraction; filtration (olives); centrifugation (olive oil)	5 nM-10 μM (0.267 nM, 180 s)	48
primicarb	SWV	laccase-modified CPE (MWCNT+paraffin oil)	vegetables (tomato, lettuce)	chopping; homogenization; QuEChERS extraction	0.99-11.5 μM (0.18 μM, 60 min)	49
carbofuran (1), carbaryl (2), formetanate (3), pirimicarb (4), ziram (5)	SWV	graphene-doped CPE surface modified with laccase and Prussian blue (graphite powder+paraffin oil)	vegetables (tomato, potato)	chopping; homogenization; QuEChERS extraction	1: 0.498-5.88 μM (0.1 μM, 15 min) 2: 0.074-0.847 μM (4.97 nM, 15 min) 3: 0.249-4.76 μM (0.059 μM, 15 min) 4: 0.299-5.66 μM (0.029 μM, 15 min) 5: 0.025-0.566 μM (5.2 nM, 15 min)	50
zearalenone (1), α-zearalenol (2), β-zearalenol (3)	amperom. (detector in CE)	unm. CPE (graphite powder+paraffin oil)	maize flour	supercritical fluid extraction with CO_2-methanol system; adsorption cartridge cleanup	1: 0.24-1.57 μM (72 nM, ---) 2: 0.21-1.57 μM (63 nM, ---) 3: 0.37-1.57 μM (110 nM, ---)	51
brevetoxin B	SWV	magnetic CPE (graphite powder+paraffin oil)	seafood	tissue disrupting with dimethyl sulphoxide (50%, w/v); centrifugation; filtration	1.12 pM-11.2 nM (1.12 pM, 30 min)	52
citrinin	amperom.	horseradish peroxidase and ferrocene-modified CPE (MWCNT+mineral oil)	rice	extraction with ACN/4% KCl (9:1); acidification; filtration; purification with n-heptane; multistep extraction with chloroform	1-11.6 nM (0.25 nM, ---)	53
tetracycline (1), oxytetracycline (2), chlortetracycline (3), doxycycline (4)	ameprom. (detector in ME)	cellulose-dsDNA-modified CPE (graphite powder+mineral oil)	beef meat	matrix solid phase dispersion extraction	1: 8.2-500 nM (4.3 nM, ---) 2: 7.5-500 nM (1.5 nM, ---) 3: 9.1-500 nM (1.9 nM, ---) 4: 10.3-380 nM (2.1 nM, ---)	54
sulfamethoxazole	potentiom.	CPE modified with molecularly imprinted polymer and sodium tetraphenyl borate (graphite powder+paraffin oil)	milk, egg / fish	homogenization; dilution with buffer; homogenization again; centrifugation / multistep extraction procedure	0.06 μM-3.1 mM (3.5 nM, <10 s)	55

Table 1 (continued)

phenolic xenoestrogens (pentachlorophenol(1), bisphenol-A(2), 2,4-dichlorophenol(3), 4-tert-octylphenol(4), 4-nonylphenol(5))	amperom. (detector in CEC)	unm. CPE (graphite powder+mineral oil)	egg, milk powder	matrix solid phase dispersion extraction	1: 1.88-37.5 μM (0.188 μM, ---) 2: 0.219-219 μM (21.9 nM, ---) 3: 0.307-307 μM (12.3 nM, ---) 4: 0.485-194 μM (48.5 nM, ---) 5: 0.454-90.8 μM (90.8 nM, ---)	56

Abbreviations used in the table (A−S): --- − not available / not found / not specified; amperom. – amperometry; acc. time – accumulation time; AdASV – adsorptive anodic stripping voltammetry; AdCSV – adsorptive cathodic stripping voltammetry; AdSV – adsorptive stripping voltammetry; ATP − adenosine triphosphate; CE – capillary electrophoresis; CEC – capillary electrochromatography; CNF – carbon nanofiber; CNT – carbon nanotube; CPE – carbon paste electrode; DP – differential pulse; DPV – differential pulse voltammetry; dsDNA – double-stranded deoxyribonucleic acid; LOD – limit of detection; ME – microchip electrophoresis; MWCNT – multi-walled carbon nanotube; potentiom. – potentiometry; resp. time – response time; SI-LOV – sequential injection lab-on-valve; SW – squarewave; SWCNT – single-walled carbon nanotube; SWV – squarewave voltammetry.

Applied electroanalytical approaches

In general, all types of voltammetric/ amperometric, coulometric and potentiometric methods can be applied with CPEs.

Voltammetry, especially differential pulse voltammetry (DPV)[23-27,29,31,34,36-38,42,45-47] and squarewave voltammetry (SWV),[28,33,41,43,48-50,52] is very often used in quantitative determinations. If lower detection limits are needed, different approaches could be applied (e.g. adsorptive, pre-electrolytic or chemical preconcentration approaches). For the determination of organic compounds in different foodstuffs the adsorptive stripping procedures are the most frequently used.[22,29-33,35,36,39-42,45-56] These procedures often improve the analytical selectivity as well.

Amperometry is also frequently applied with CPEs. On one hand, enzyme[22,32,53] or different nanoparticle and nanofiber-modified[21,35] amperometric sensors were applied in chronoamperometric determinations. On second hand, amperometric detectors based on unmodified[51,56] and modified carbon pastes[30,54] were successfully used in sequential injection lab-on-valve (SI-LOV)[30] system, capillary and microchip electrophoresis (CE and ME),[51,54] and capillary electrochromatography (CEC)[56] for the determination of very different analytes.

CPEs can also be used as potentiometric sensors. Ion exchanger[39,40] and molecularly imprinted polymer (MIP)-based[55] potentiometric sensors were successfully applied in direct potentiometry and potentiometric titrations.

Applied electrode types

While unmodified CPEs are still in use in determination of organic compounds in food samples,[42,51,56] most papers are dealing with the application of different chemically or biologically modified electrodes.[21-41,43-50,52-55] Concerning the basic constituents of the CPEs, combination of graphite powders and mineral (paraffin) oil/grease-type binders is still the mostly applied.[21,22,24-31,33,35-38,41-43,45-47,50-52,54-56] Newer or atypical binders include different ionic liquids, dioctyl phthalate and tris(2-ethylhexyl) phosphate.[27,28,39,40,43] The use of single-walled and multi-walled carbon nanotubes (SWCNTs, MWCNTs), carbon nanofibers (CNFs) and graphene − even as the main paste constituent or just a modifier − is very frequent.[22-24,26,27,32,33,35,37,41,43,49,50,53] Surface and bulk modification with different (micro/nano)particles and composites (Au, ZnO, NiO, MnO_2, TiO_2, Ca-montmorillonite, Al-doped SiO_2, ZnO/CNT, Pt/MWCNT, etc.) is quite usual.[21,25,27-29,31,33,36,38,41,43,44] Application of molecularly imprinted polymers − as unique modifiers for selectivity enhancement both in voltammetric and potentiometric measurements – became also usual.[45-47,55] Some special approaches like a magnetic CPE and a competitive double-surface assay were also described for the determination of selected organic analytes in food samples.[33,52]

CONCLUSIONS

It can be concluded that the use of CPEs for the analysis of organic compounds in foods is still very

frequent and attractive. On the other hand, different chemical and biological modification procedures, miniaturization, coupling with separation techniques and microfluidics, but portability and low cost also, will assure an important position for them in future food analysis too. Hopefully, this review article will contribute to the further popularization and wider use of the often underestimated CPEs in analytical chemistry.

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