Biosensors and Bioelectronics 146 (2019) 111758

Contents lists available at ScienceDirect

Biosensors and Bioelectronics

journal homepage: http://www.elsevier.com/locate/bios

Disposable electrodes from waste materials and renewable sources for (bio) electroanalytical applications

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ARTICLE INFO

Keywords:
Disposable electrodes
Screen-printed electrodes
Paper-based electrodes
CDtrodes
Recycled electrodes
e-waste

ABSTRACT:

The numerous advantages of disposable and screen-printed electrodes (SPEs) particularly in terms of portability, sensibility, sensitivity and low-cost led to the massive application of these electroanalytical devices. To limit the electronic waste and recover precious materials, new recycling processes were developed together with alternative SPEs fabrication procedures based on renewable, biocompatible sources or waste materials, such as paper, agricultural byproducts or spent batteries. The increased interest in the use of eco-friendly materials for electronics has given rise to a new generation of highly performing green modifiers. From paper based electrodes to disposable electrodes obtained from CD/DVD, in the last decades considerable efforts were devoted to reuse and recycle in the field of electrochemistry. Here an overview of recycled and recyclable disposable electrodes, sustainable electrode modifiers and alternative fabrication processes is proposed aiming to provide meaningful examples to redesign the world of disposable electrodes.

1. Introduction

Disposable electrodes were widely applied in the development of portable electroanalytical devices in the last decades (Killard, 2017). Their application in point-of-care diagnostics and in-situ analytical monitoring is groundbreaking in biomedical, pharmaceutical (Mohamed, 2016), industrial, cultural heritage (Micheli et al., 2018) food and environmental safety (Li et al., 2017, 2012) related fields. The disposable nature of these electrodes is often associated with important characteristics such as portability, low-cost, ease of use and mass production (Thiyagarajan et al., 2014). These characteristics led to a revolution in the design and applicability of electrochemical sensors, modifying deeply the materials, the fabrication methods and the technologies behind their conception. Nowadays, thanks to disposable electrodes, it is possible to monitor glucose in blood as well as mustard gas with wearable origami sensors, to quantify drugs of abuse in the airports or water contaminants directly at riversides.

Despite all these advantages, the massive use of these devices is producing increasing quantities of composite solid waste difficult to treat or recycle. Disposable electrodes will be soon counted among the devices contributing to the *electronic waste*, also indicated as e-waste. This global phenomenon commonly linked to discarded electronics, such as smart-phones, computers and televisions, concerns also small-portable devices. Since the beginning of the twenty-first century, e-waste has been recognized as an urgent environmental, health-related and economic issue particularly in developing countries with an estimate global annual growth of 8% (Amankwah-Amoah, 2016; Perkins et al., 2014). To face this issue, sustainable and affordable recovering and recycling treatments are needed together with the development of a new generation of biodegradable electronics. Therefore, also the world of disposable electrodes needs to be redesigned following the guidelines of sustainability to become an essential example of *green analytical chemistry,* as suggested by Yáñez-Sedeño et al. (2019). Disposability should be achieved with non-toxic materials, eco-friendly solvents and reagents, with a design that minimize the amount of waste, considering each step of the electrode life-time, from production to disposal. Even

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https://doi.org/10.1016/j.bios.2019.111758
Received 1 August 2019; Received in revised form 23 September 2019; Accepted 1 October 2019
Available online 7 October 2019

though at present only few examples of completely recyclable disposable electrodes have been reported, there is a growing interest towards the use of waste materials as substrates for screen-printed electronics, the combination of high performing nanocellulose within biocompostable devices and the optimization of greener path to synthesize electrode modifiers.

Trying to address this interest, the present review proposes an overview of renewable and waste-based disposable electrodes. From paper based electrodes to the first true historical example of disposable electrodes based on e-waste (CD/DVD electrodes) to the more recent examples of commercial SPEs recycling and re-use, this review aims to be a guidebook, providing meaningful examples on each topic and helping the research community to redesign the world of disposable sensors and biosensors, towards a greener approach to (bio)analytical electrochemistry.

In this outline, the materials structure and properties, the fabrication strategies and the final applicability of the devices will be emphasized, particularly for the most recent examples. To suggest novel applications and further discuss all the issues related to e-waste recycling and sustainable approaches in electronics conception, a section dedicated to green modifiers and recycling treatments applicable to disposable electrodes is included. From this last part, the necessity to integrate different research fields clearly emerges: from the synthesis of nanomaterials using biomass to the development of green electrode modification protocols with a focus on circular productions.

2. Paper-based disposable electrodes

Paper is the most widely used material for the development of disposable sensors. Initially, microfluidic paper-based electrochemical sensors, known as EμPADs or μPEDs, were designed to combine paper-based microfluidic analytical devices (μPADs) within electrochemical sensing tools. Paper-based electrodes suddenly showed many other advantages related to: (1) the recyclable and inexpensive nature of the paper substrate, (2) the ease of functionalization and integration with printing systems, (3) the adaptability of their design and (4) the numerous properties of cellulose and its derivatives, from microfibers to nanocellulose. The first examples of disposable filter paper oxygen electrodes in the late '90s (Suzuki, 1994; Yang et al., 1997) gave rise to several types of paper-based electrochemical sensing strategies from pencil drawn electrodes to recycled paperboard SPEs. The growing interest in hybrid analytical tools together with the fast development of new production technologies have further increased the variety of paper-based electrodes in the last decade. Even though these electrodes cannot be strictly divided in several classes, in the next paragraphs five main typologies (μPEDs, SPEs on paper, pencil drawn electrodes, SPEs with cellulose derivatives and recycled paper electrodes) were identified to guide the reader in a general overview focusing on the fabrication processes, performances and applicability of these biocompatible disposable electrodes. The application of cellulose derivatives in the design of highly performing substrates, organic solvent-free inks and electrode surface modifiers will be described to address the increasing interest in versatile and renewable materials for green electronics. Moreover, to show how disposable electrodes can be more sustainable, key-examples of SPEs fabricated from recycled paper with zero-waste processes were selected among the emerging generation of recycled material-based SPEs.

2.1. μPEDs

Pulp papermaking processes allow to easily tune the physical properties of paper (i.e. thickness, porosity, flexibility, permeability, etc.) making this material extremely versatile and attractive for analytical applications, particularly paper-based sensors (Liana et al., 2012). The development of photolithography, wax printing and chemical vapor-phase deposition techniques allowed drawing arrangements of hydrophobic/hydrophilic microstructures. . In these networks, the transport of small volumes of liquids (few nanoliters) occurs spontaneously by capillary action and the dimensions of the channels (between 100 nm and 100 μm) allow the molecules to diffuse faster according to the principles of microfluidics (Convery and Gadegaard, 2019). Since their introduction in the market in 2007, these paper-based microfluidic analytical devices (μPADs) were successfully combined with a wide range of detection strategies (Fu and Wang, 2018) for lab-on-chip and point-of-care (POC) applications. According to Yang et al. (2017), their integration within electrochemical sensors and biosensors, also known as EμPADs (here always indicated as μPEDs), is the most extensively investigated so far. Once loaded in the μPEDs paper-microfluidic pattern, the sample wicks into specific spots in direct contact with the working electrode or the receptors (i.e. antibody, enzyme, etc.) and the electrochemical signal is recorded. μPEDs were found to be particularly suitable for POC diagnostics (Lim et al., 2017) and environmental analysis (Almeida et al., 2018) because of their: (1) sensitivity, selectivity and reproducibility; (2) flexibility, lightweight, portability and adaptable design; (3) low-cost and eco-friendly components; (4) ease of use. In 2009, Dungchai et al. (2009) reported the first example of a μPEDs assay achieving the simultaneous determination of uric acid, glucose and lactate in biological fluids. By drawing hydrophilic trilobed areas on filter paper through photolithography, they assure the homogenous diffusion of the sample volume to the three SPEs in the lobes. Then, by screen-printing the working electrodes (WEs) with a carbon ink doped with Prussian Blue and modifying them with different oxidase enzymes, they achieved sensitive and selective amperometric responses. The μPEDs reported by Kumachev et al. (2010) were characterized by a simpler design and applied in proof-of-concept studies for the quantification of glucose in artificial urine using glucose oxidase and for the selective determination of Pb(II) in an aqueous solution containing a mixtures of Pb(II) and Zn(II). This latter part of the study clearly showed the role of paper microfluidics. Indeed, the continuous wicking of sample through the paper substrate allowed to increase the amount of metal deposited during anodic stripping voltammetry, thus the sensitivity of the sensor. These first proof-of-concept studies were soon integrated with commercially available instruments, such as glucometers (Nie et al., 2010), for rapid and quantitative electrochemical analysis.

From 2011 on, μPEDs performances and fabrication processes were highly improved by substituting photolithography with wax-screen-printing. This technique allowed lowering the background reactivity, obtaining optimal results in terms of stability and reproducibility (Chailapakul et al., 2011). Wax-screen-printing was successfully applied also to 3D microfluidic paper-based electrochemical immunodevices (3D-μPEIDs), such as the one reported in Fig. 1. Designed by Zang et al. (2012), this 3D-μPEID allowed the simultaneous monitoring of four cancer marker antigens. Its selectivity depends on a sandwich architecture, made of a primary antibody immobilized on the paper-electrode surface, an antigen and a secondary antibody labelled with horseradish peroxidase, HRP. The binding event took place in the inner 3D-structure of the sensor and is followed via HRP-*o*-phenylenediamine-H_2O_2 electrochemistry with Differential Pulse Voltammetry (DPV). A further signal amplification was achieved by modifying the working electrodes with highly conductive MWCNTs. Despite their performances and versatile design, 3D-μPEIDs immunosensors were often considered too complex and unstable for routine analysis and not suitable for technological transfer.

Since 2013, the possibility to draw graphite pencil electrodes (PDEs), discussed latter in paragraph 1.3, on μPADs designing two-electrode systems for glucose monitoring appeared to be a suitable alternative to the corresponding biosensors (Santhiago and Kubota, 2013). Notwithstanding the composite nature of 3D-μPEDs and PDE-μPADs, the minimal quantities of nanomodifiers and inks required and the biocompatible nature of the substrates used ensure their easy disposal, compared to more traditional plastic or ceramic based substrates.

Apart from POC and clinical applications (Fu and Wang, 2018; Lu

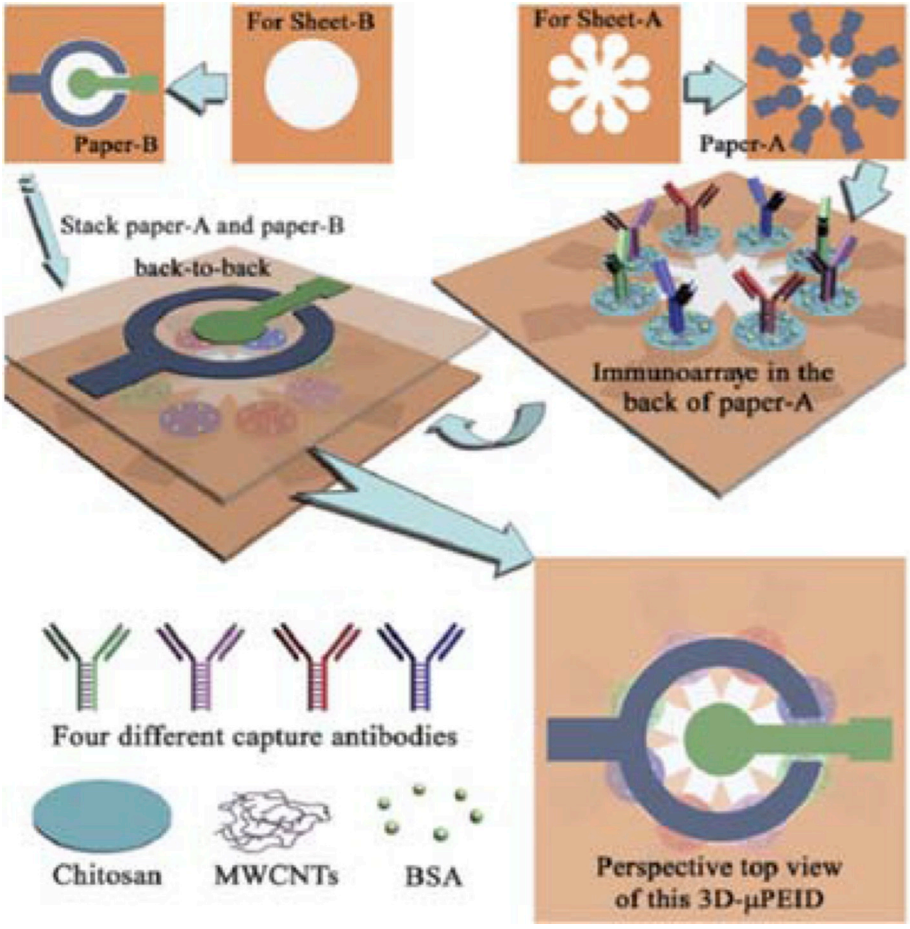

Fig. 1. Scheme of fabrication of a 3D-μPEID reported by (Zang et al., 2012) consisting of two sheets (showed on top). The sheet-A hosted eight screen printed WEs (two for each cancer marker antigen) modified with chitosan/MWCNTs, four different primary antibodies and Bovine Serum Album (BSA). The sheet-B presented the WE connection printed with carbon ink and the reference electrode printed with an Ag/AgCl ink. In the final 3D arrangement, the sheet-B was placed on top of sheet-A, as showed in the right bottom corner.

et al., 2012; Santhiago and Kubota, 2013; Wang et al., 2016), eco-friendly reagentless μPEDs were recently used in the determination of numerous contaminants (from inorganic to organic and biological ones) related to food and environmental safety. Cinti et al. (2016) reported a simple sensing strategy for the detection of phosphate in water. By loading molybdate ions in the paper, the authors were able to deduce phosphate concentration from the voltammetric response of the phosphomolybdic complex formed in the substrate matrix. The inter-electrode reproducibility of these μPEDs was guaranteed by the stability of the paper characteristics and the simplicity of the modification protocol. Bhardwaj et al. (2017) designed a bio-conjugate to detect *Staphylococcus aureus* and prevent foodborne diseases. In this immunosensor, the antibodies were covalently bound to single walled carbon nanotubes (SWCNTs). The formation of the antigen-antibody complex was followed by DPV in presence of a redox mediator. Increasing peak currents were recorded at higher bacteria concentrations. Indeed, the complex formation appears to change the steric hindrance of the bioreceptor and to help the interaction of the redox mediator with the SWCNTs surface.

The examples provided clearly show the high versatility of these integrated devices that can be easily combined with a wide range of electrochemical techniques (chronoamperometry, CV, DPV, etc.) and even with electrochemical impedance spectroscopy (EIS) (Ruecha et al., 2019) and electrochemiluminescence (Ge et al., 2012). All these advantages have contributed to the development of fully optimized and wearable/portable μPEDs and promising researches are still ongoing (Amor-Gutiérrez et al., 2019; Fan et al., 2019). In particular, Cao et al. (2019) recently reported a complete study on a wearable 3D-μPEIDs, optimized for the real-time monitoring of sweat metabolites (glucose). These 3D-μPEIDs take advantage of the glucose oxidase activity to assure a selective response (glucose oxidation) with a lower detectable concentration of 5 μM even during on-body tests. The enzymatic bioreceptor, loaded in a polymeric layer at the working electrode surface, is free to interact with the target molecule and the paper permeability assures the control of the modification. Another key-example is the 3D-μPEID coupled with a contactless conductivity detection strategy that was optimized for the monitoring of *E. coli* cells (Duarte et al., 2019). By measuring the current density, this sensor allows to determine the cellular population with a one-step protocol, without the need of specific labels and requiring only nanoliters sample droplets. Similar 3D-μPEIDs could represent a low-cost and ecological alternative to the traditional cell counting methods used in biomedical and biological research fields.

Instead of printing the electrodes on the μPADs paper, it is also possible to add paper disks directly on commercial disposable electrodes to gain all the advantages of droplet-based microfluidics (Chandra Sekar et al., 2014; Kuek Lawrence et al., 2014). One of the first examples was provided by Yang et al. (2014). They reported an amperometric glucose biosensor in which pure cellulose paper pads, loaded with glucose oxidase, lied on top of SPE previously modified with platinum nanoparticles (PtNPs). μPADs and filter paper were largely used as biocompatible and versatile electrode modifiers in paper disk screen printed electrodes. Filter paper disks loaded with specific reagents were juxtaposed on the SPEs for the sensing of lead and mercury in water (Fang et al., 2011; Sánchez-Calvo et al., 2019; Tan et al., 2010), acetylcholinesterase in biological fluids (Panraksa et al., 2018), glucose in blood (Chandra Sekar et al., 2014). As underlined by Moreira et al. (2018), the paper matrix is a particularly *adequate microenvironment* to follow the direct electrochemical behaviour of enzymes. Indeed, the inner structure of paper, mainly filter paper, allows to absorb different kind of reagents, from

biomolecules to metals and nanomaterials, without altering their properties and providing highly reproducible modifications. Sánchez-Calvo et al. (2019) recently reported a selective sensor for Hg (II), by modifying the paper disk with different hybrids of gold nanoparticles and carbon nanofibers or reduced graphene oxide. This strategy makes only the paper disks disposable while the SPEs can be reused after an accurate washing treatment limiting the solid waste production. Moreover, paper disk can be easily combined with already modified SPEs electrodes. This is the case of a phenylalanine biosensor (Moreira et al., 2018), in which reduced graphene oxide modified screen-printed electrodes were combined with paper disks loaded with phenylalanine dehydrogenase, as depicted in Fig. 2.

Paper disks can also be used to load the sample and assure an improved contact with the electrode surface. For example, Kong et al. (2014) designed an assay for the screening of glucose in whole blood by modifying the electrode surface with a biocomposite and loading the sample on paper disks before laying them on the SPEs. The biocomposite consisting of graphene, polyaniline, gold nanoparticles and glucose oxidase enhanced the sensitivity of the sensor making real whole blood sample analysis possible. Paper disks as well as other type of μPADs modifiers combined with screen-printed electrodes are here considered as μPEDs because their performances strongly depend on the microfluidic properties of paper. Despite their outstanding performances, most μPEDs are not yet ready for commercialization. As pointed out by Akyazi et al. (2018), in many cases the manufacturing process still needs to be streamlined and the integration with flow control methods is often missing. This last point is particularly important for commercial applications: often the flow control of the sample in the μPEDs is still controlled by external devices and thus not easily employable on-site.

2.2. Paper as electrode substrate

Apart for its microfluidic properties, paper was found to be a suitable substrate for its high compatibility with inkjet (Moya et al., 2017) and screen printing (Renedo et al., 2007) techniques and the ease of combining it with different surface modifiers, from polymers and enzymes (Kit-Anan et al., 2012; Tao et al., 2016) to nanomaterials and redox mediators (Nantaphol et al., 2019). All these advantages together with the possibility to easily integrate these electrodes within impedimetric (Ihalainen et al., 2013), potentiometric (Sjöberg et al., 2016) and voltammetric (Määttänen et al., 2013) detection strategies have led to the rapid development of sensors on paper, generally referred to as electrochemical paper-based analytical devices (ePADs).

In the ePADs, filter paper is often substituted by commercial or multilayer-coated recyclable paper. The choice of the paper is fundamental, especially when molecular recognition layers has to be immobilized on its surface. Thanks to the rich surface chemistry of cellulose-based materials, it is possible to immobilize bio receptors by simple physisorption (Kannan et al., 2015) or with bio-affinity interaction (biotin-avidin, Protein A/G, etc.) (Credou and Berthelot, 2014) or by covalent bond, with a variety of chemical reactions (maleimide cycloaddition, active esterification, click chemistry, etc.) (Böhm et al., 2018; Hong et al., 2018). All these strategies, developed mostly for paper-based microfluidic devices, have to be adapted to paper based electrodes, considering also the presence of the electrode materials (conductive inks, nanoparticles, etc.) on the paper substrate. The in-depth characterization of these interaction (electrode-paper substrate-bioreceptor) is still an open challenge and should be carefully considered to improve reproducibility and to address the problem of scalability of the proposed biosensors.

Maattanen et al. (Määttänen et al., 2013) reported a complete study in which the design of a paper-electrochemical platform was largely tested in presence of different modification from self-assembled monolayers and polyaniline to poly(3,4-ethylenedioxythiophene) (PEDOT) showing promising results. PEDOT doped with poly(styrene sulfonate) was inkjet-printed on paper in order to make electrocardiographic devices (Bihar et al., 2017). To develop a sensor for ssDNA and dsDNA detection in biological fluids, Cinti et al. (2018) tested both filter and copy paper, as substrates for carbon SPE modified with gold nanoparticles covalently bound to methylene blue-tagged oligonucleotides probes, with excellent results. In this kind of POC applications the flexible nature of paper plays a fundamental role, as well as its chemical nature that makes it particularly suitable also to test newly developed printing inks (for an overview see (Chu et al., 2017)). The possibility to load the reagents in the paper substrate previously described in disk-paper SPEs (par.2.1) was exploited also in the design of wearable SPE ePAD; one of the last examples is the enzymatic origami sensor designed for mustard gas detection designed by Colozza et al. (2019).

At present, the evolution of screen printed electrodes technologies is going towards a progressive simplification of their fabrication process

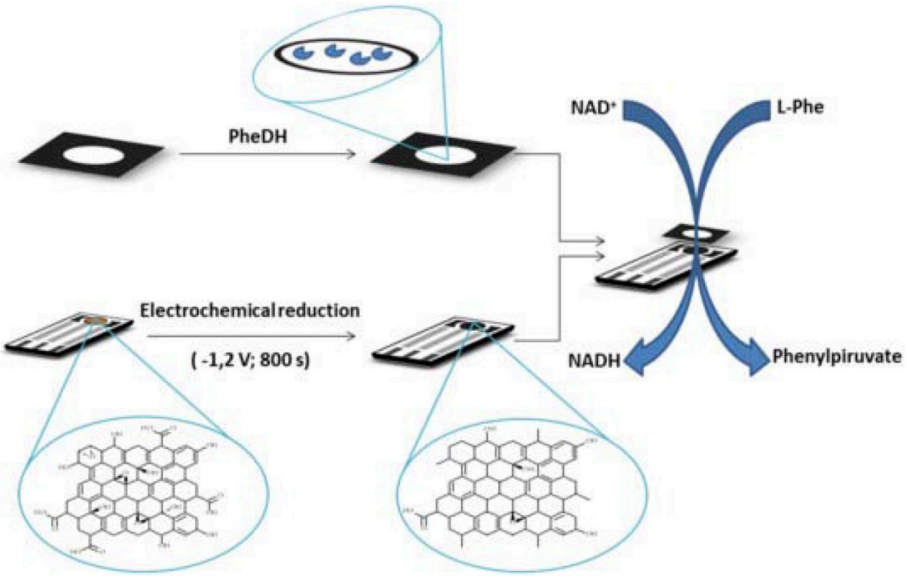

Fig. 2. Schematic representation of the paper disk electrochemical sensor for the quantitative determination of phenylalanine in neonatal samples, reported by Moreira et al. (Moreira et al., 2018). In this device, the carbon screen-printed electrode modified with electrochemically reduced graphene oxide was combined with a paper disk previously loaded with phenylalanine dehydrogenase.

and materials. By substituting the wax printer with an inexpensive cutter printer, De Oliveira et al. (de Oliveira et al., 2019) produced ePADs with two electrochemical cell compartments for multiplexed analysis of drugs. The strength of this fabrication process consists in the possibility to define the filter paper wettable areas simply attaching a low track transfer tape without the need of wax printing and to cut the electrodes arrangement pattern before depositing the inks. It is important to note that different paper substrates may have different recycling process (R. McKinney, 1995) and the trade-off between surface characteristics (i.e. porosity, sizing agents, thickness, pulp composition) and recyclability has to be carefully evaluated.

2.3. Pencil drawn electrodes on paper

Together with polyester and PVC (Bernalte et al., 2016; Foster et al., 2016; Honeychurch, 2015), paper represents the most common substrate used in the creation of pencil drawn electrodes (PDEs) for electroanalytical applications (David et al., 2017; Torrinha et al., 2018). Since the first examples of PDEs published in the 50′-60′, the pencil-drawn approach showed many advantages from cost-effectiveness and accessibility to the possibility of a rapid prototyping of complete sensing platforms (Dossi et al., 2016; Li et al., 2016a). Most PDEs were drawn from readily available pencils, particularly pencil leads that consist of a mixture of graphite, clay and wax. The impact of the different fractions of graphite and additives in the mixture was largely investigated (Dossi et al., 2014) along with the quality of the paper substrate and its preparation (Dias et al., 2018). For example, Li et al. (2016a) chose a chromatographic filter paper substrate to develop a PDE for dopamine. Before drawing the electrodes, the substrate underwent a pre-printing step and a heat treatment to assure reproducibility and inter-electrode consistency of the three-electrode pattern. Filter paper was used also for micro-PDE, where Prussian blue was electrodeposited at the micro-working electrode to measure hydrogen peroxide (Jomma et al., 2017). Similar PDEs fabricated using computer-controlled mechanical plotter/cutter and drawing system could easily enter the market and be applied on a large scale. Dossi et al. (2017) reported a procedure to assemble fully drawn PDEs, describing its optimization and the numerous parameters that need to be defined (i.e. pencil pressure, contact time, etc.). Commercial A4 size paper was used to draw inexpensive electrodes using a carbon powder nail polish ink (Pradela-Filho et al., 2017); the obtained PDEs were then successfully tested for the detection of dopamine. Li et al. (2016b) developed an enzymatic glucose sensor on origami paper. The three-electrode detection zone was put in contact with the enzyme zone (previously filled with the bioreceptors) by folding the origami paper before adding the sample drop. From cellulose pulp is also possible to obtain more resistant substrates often required in PDE devices, such as the corrugated fiberboard used in a catechol sensor (Orzari et al., 2018).

However, among the other possible materials for PDE substrates, paper is still the most inexpensive, easy to adapt, biocompatible and recyclable one. Its application in the so-called “Do It Yourself” sensors keeps increasing and was extended also to other electronics (Dossi et al., 2016), such as fuel cells or photodiodes. Moreover, paper-based PDE can be integrated within original configurations and coupled with other devices to tackle multiple analytes (Dossi et al., 2018).

2.4. Cellulose derivatives in SPEs

The wide application of nanotechnologies in the material science is leading to the rapid development of highly performing derivatives of well-known materials, such as cellulose (He et al., 2018) particularly suitable to redesign electronics, as showcased by Agate et al. and Hajian et al. (Agate et al., 2018; Hajian et al., 2019). Cellulose is a complex biopolymer: it consists of six hydroxyl groups within the repeating structure of β-d-glucopyranose units which can be chemically treated to obtain micro or nanofibrillated cellulose, nanocrystalline cellulose (NCC), etc. (for a complete overview see (Ergun et al., 2016)). The different properties of cellulose derived materials have led to higher compatibility with printed layers in terms of (1) wetting, adhesion and drying, (2) controlled surfaces characteristics and (3) improved performances and sustainability in the final devices. The advent of organic electronics has further increased the interest in cellulose derivatives. In 2018, Li et al. (Y. Li et al., 2018b) obtained sustainable SPEs by inkjet printing of a water-based carbon ink on biocompostable nanocellulose substrates. Moreover cellulose derivatives showed unexpected properties: transparent, conductive and printable nanocomposites consisting of cellulose nanofibrils (CNF) modified with TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl) and carbon nanotubes were designed by Nogi et al. (2013) as alternative substrates to traditional polymeric one. Thanks to its properties, this material is largely applicable in electronics and optoelectronic. The same functionalized CNF were combined recently with micropatterns of silver nanowires (Kim et al., 2018) for the fabrication of highly bendable conductive microelectrodes.

Micro/nanocomposite cellulosic materials were specifically optimized by Torvinen et al. (2012) to substitute oil-based plastic in SPE applications. Because of their stability and low surface roughness, these substrates were more performing than normal cellulose. The optimal formulation for this material was obtained by screening different combination of microfibrilled cellulose (MFC) and inorganic fillers and providing a full characterization of their physical properties. A wide range of combinations was tested, not only in the development of electronic substrates (Gaspar et al., 2014), but also for organic solvent-free inks (Barras et al., 2017). Low percentages of MFC together with carboxymethyl cellulose (about 1% and 2%, respectively) were used as binders for *green water-based SPE inks* to be applied in lithium ion batteries (El Baradai et al., 2016). Recently, El Baradai et al. (El Baradai et al., 2018) tested also cellulose nanofibers for the same application, observing electrochemical properties and cycling stability equivalent to those obtained using conventional organic solvents and fluorinated binders. CNF were also often combined with silver-based inks: in 2017, Hoeng et al. (2017a) optimized a cellulose nanocrystals-silver nanoparticles inks and applied it to the printing of flexible and sustainable electronics on paperboard. The authors tested also silver nanowires-CNF inks, investigating the thickening role of CNF and its ability to provide a controllable viscosity to the screen printing suspension (Hoeng et al., 2017b). These inks, first designed for energy storage devices (in the fabrication of electrodes, separators, etc.), are finding wide application in electronics, from conductive coatings (Kumar et al., 2017) to self-standing conductive adhesives (Edberg et al., 2017). However, the performances of these cellulose-based inks are not yet comparable with the traditional ones, particularly in terms of conductivity and stability, but these drawbacks can be solved by increasing the loading of metal nanoparticles or minimize the insulating fractions of cellulose.

In general, the versatility of these derived materials depends on the possibility to adjust their surface and structural properties (i.e. porosity, smoothness, wettability and density) to functionalize the fibres and to combine them with nanomaterials or other modifiers, such as conductive polymers (Dias et al., 2019). Apart from their attractive technological applications, cellulose derivatives based SPEs have also enabled to tackle plant bioelectricity and perform fundamental studies on their physiology (Keller et al., 2017).

2.5. From recyclable to recycled paper substrates

Apart for its versatility, microfluidic properties and all the above mentioned advantages, paper was often chosen simply because is an easily recyclable substrate. Ideally, most of the paper-based sensing devices described above could be fabricated from recycled paper by adjusting their manufacturing procedures. Despite the numerous examples showing how recycled paper can be used in electronic applications, from double-layer capacitors (Kalpana et al., 2009) to humidity sensors (Mraović et al., 2014), there are still only few examples of

functioning SPEs from recycled paper (de Araujo et al., 2017; Mianehrow et al., 2017). Particularly interesting in our overview is the one reported by De Araujo et al. (de Araujo et al., 2017), in which a single-step laser scribing process was designed for the fabrication of nanostructured electrodes from waste paperboard. By combining a green production process with a reproducible technology (Fig. 3), the authors were able to obtain SPEs with very good performances and tested them for pharmaceutical, clinical, food, and forensic analytical applications. This technology was developed considering that the pyrolysis of organic compounds generates carbon materials. Therefore, it is possible to produce conductive porous non-graphitizing carbon material by CO_2 laser pyrolysis of suitable precursor materials, as paperboard.

This last example of green SPEs production from waste sources suggests the strong potentialities of this production process. Even though paper was one of the most investigated materials for SPEs productions, there are also other recycled substrate to be considered. Looking at the overall life cycle of SPEs, paper-electrodes should be further improved and combined with recycled electrodes modifier (from electronic waste as well as from biomass) or designed to be compostable with easy protocols.

3. Recycled electrodes material

With the increasing rise of electronic wastes the research community tried to find ways of reusing and recycling such waste, commonly composed of noble metals and other materials, attractive for electroanalytical applications (Ding et al., 2019). One very successful protocol, used for the first time in the early 2000s (Angnes et al., 2000), allows to obtain Au and Ag disposable electrodes from compact discs (CD) and digital versatile disc (DVD); the quality of the manufacturing of these supports assure a very high reproducibility and homogeneity in the obtained electrodes. Also other waste materials were used more recently to realize electrodes or surface modifiers like copper nanoparticles from electric cables (Abdelbasir et al., 2018) or carbon rod electrodes from spent zinc-carbon batteries (Honeychurch, 2019).

3.1. Disposable electrodes from CD/DVD

CDs and DVDs are compact disc data storage devices developed in the early '80s and '90s (Immink, 1998). They are composed by a polycarbonate layer, in which the data are encoded, covered by a thin layer of aluminium, gold or silver, that reflects the laser light of the reader, and a protective polymeric coating. CD data are represented as tiny indentations known as "pits", encoded in a spiral track moulded into the top of the polycarbonate layer. The areas between pits are known as "lands". Each pit is approximately 100 nm deep by 500 nm wide, and varies from 850 nm to 3.5 μm in length. The distance between the tracks is 1.6 μm (Cope, 1993; Baert et al., 1992). The difference between CDs and DVDs lies in the spacing of the indentation: DVDs tracks are around 800 nm apart with a width of 300 nm, thus allowing to store more data.

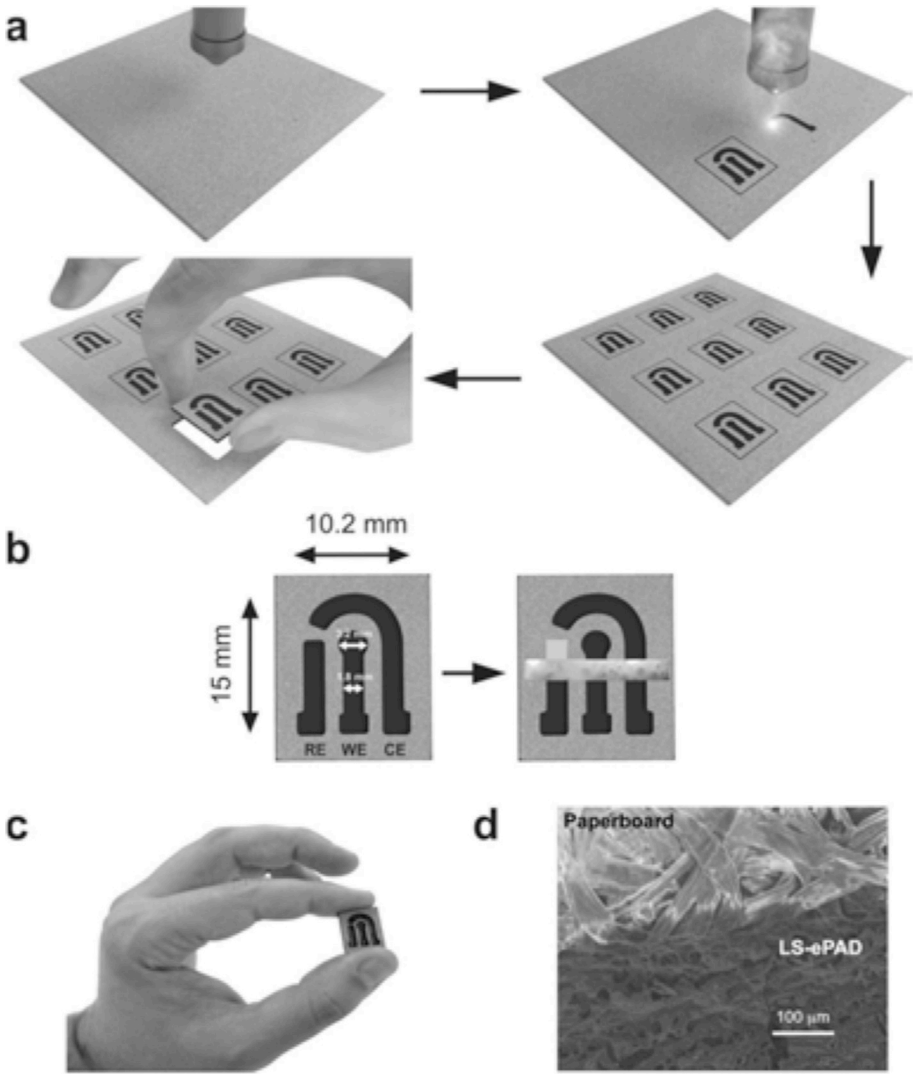

Fig. 3. Scheme of the SPEs production reported from (de Araujo et al., 2017). a) Representation of the fabrication process. b) Dimensions of the SPEs and the working, counter, and reference electrodes (WE, CE, and RE, respectively). Also shown is the patterning step (area delimitation using Cascola glue) and silver ink painting processes on the top of the RE scribed carbon film. c) The dimensions of SPEs in manual handling. d) Scanning electron microscopy (SEM) image of the interface between the paperboard and the non-graphitizing carbon film formed by CO_2 laser radiation.

CDs and DVS are commonly available and relatively cheap compared to traditional electrode materials. In 2000, Angnes et al. realized the potentialities of these electronic waste materials and developed a protocol to obtain disposable gold electrodes (Angnes et al., 2000). After "slicing" the CDs, they removed the protective coating with concentrated nitric acid (as showed in Fig. 4), and assembled the electrode, defining the working electrode area.

The CDtrodes showed performances comparable to macro Au electrodes both for the stripping analysis of copper ions and in a flow system setup. They also proved the remarkable versatility of the proposed protocol, obtaining electrodes with a geometric area from 10^{-6} cm^2–100 cm^2. Following this influential publication, other research groups started employing these disposable electrode for a variety of analytical applications. Several techniques were developed to obtain different architectures: the surface of the CD, after removing the polymer layer, can be patterned by means of laser printing (Daniel and Gutz, 2003a), inkjet masking (Lowinsohn et al., 2006), by heat transfer methods (Daniel and Gutz, 2005) or more recently by xurography (De Santana et al., 2012). The possibility to print different design on the surface of the compact disc was implemented: using toner mask to define the electrodes area (Lowinsohn et al., 2006), heat transferring a patterned design to obtain interdigitated electrodes (Daniel and Gutz, 2005), dual band microelectrode (Paixão et al., 2003), or even complete arrays with multiple working electrodes (Cho et al., 2007) coupled with microfluidics devices (Daniel and Gutz, 2003a; Kirkpatrick et al., 2010). Also the complete layered structures of CD and DVD can be used to fabricate innovative electrode setups. Hantezadeh and Rashid-Nadimi (2016) reported a flat redox microbattery arrays obtained by simply drilling holes in the compact disc to prepare thin band electrodes, without any previous treatment. The versatility of the fabrication approach was proven by coupling more than two microbatteries, connecting them in parallel or in series. Taking advantage of the grooves on the CDtrodes it was also possible to obtain controlled deposition of metals and other nanomodifiers, such as zirconia thin films (Yu et al., 2002). These CDtrodes were used in a variety of analytical applications both for organic and inorganic molecules with different configurations and performances, as further discussed in the next paragraphs.

3.1.1. Determination of metal ions

Since the first account from Angnes et al., these disposable Au and Ag electrodes were deemed especially suitable for heavy metals analysis. For stripping analysis of many metallic ions, gold electrodes give larger and sharper peaks than platinum or mercury electrodes, attaining lower detection limits even in real samples (Hanrahan et al., 2004). CDtrodes showed the same performances of bulk electrodes but their disposable nature avoids problems of surface fouling and subsequent time-consuming cleaning procedures between analysis. The quantification of copper ions in different media have been reported; Angnes et al. achieved the simultaneous quantification of copper and mercury (Angnes et al., 2000) by means of potentiometric stripping analysis, also in tap water and sugar cane spirit (Richter et al., 2001) attaining a low limit of detection (30 ng/L) with a very good reproducibility (less than 2% on 20 replicates). The same research group also used CDtrodes for the quantification of copper ions in lubricating oils combining an ultrasonic extraction step with square wave anodic stripping voltammetry (Munoz et al., 2006b). Walcarius and Sibottier used instead the CDtrode as a platform to support an amine-functionalized silica film. The silica film was used as a pre concentration matrix for Cu(II) ions, subsequently detected by differential pulse anodic stripping voltammetry (DPASV) (Walcarius and Sibottier, 2005). Also Daniel and Gutz exploited the gold CDtrodes as a support for TiO_2 nanoparticles for in-situ photocatalytic decomposition of organic matter. The electrode was integrated in a microfluidic device for the voltammetric quantification of Cu(II). These approaches show the remarkable versatility of the CDtrodes. More recently copper quantification was performed at sub-microband electrodes (Zhu et al., 2018). In this configuration the working electrode is obtained from the side thickness of the CD gold layer, showing performances similar to conventional microelectrodes.

The quantification of Hg(II) was performed on different types of CDtrodes as well; Agnes research group employed these electrodes for the chronopotentiometric stripping determination of mercury in natural water (Augelli et al., 2005) and urine samples, after ultrasonic digestion (Munoz et al., 2006a). Microwave digestion was instead applied to the determination of mercury in fish and shrimp samples (Augelli et al., 2007; Radulescu and Danet, 2008) and in crude oil and petroleum-based

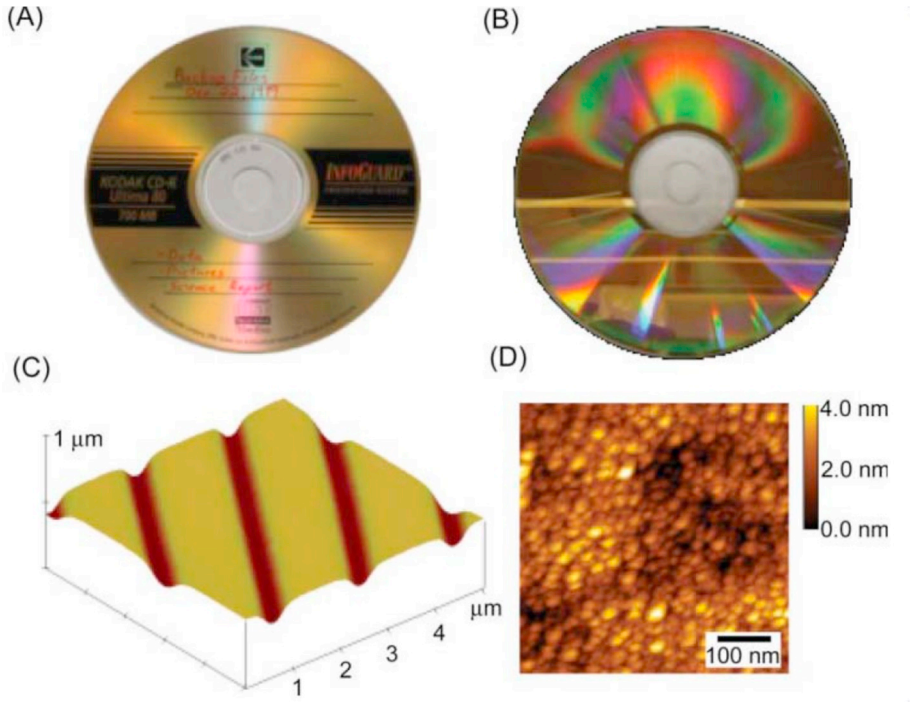

Fig. 4. Image of CD-Rs before (A) and after (B) removal of the polymer layer, and atomic force microscope. (AFM) images of the CD-R gold substrates (5 × 5 mm^2), (C), and 500 × 500 nm^2, (D); reported from (Yu, 2004).

fuels (Munoz et al., 2007). In these examples, the high quality of the CDtrodes assured good performances even for complex natural matrices.

Also Ag electrode obtained from compact disc were used in the stripping analysis of metal ions. Honeychurch (2013) reported the underpotential deposition of Pb at silver CDtrodes and its quantification by means of anodic stripping voltammetry (ASV) in spiked drainage water, with high recovery (95%) and good reproducibility (<3%). Stripping analysis of other uncommon ions was performed at CDtrodes gold electrode: Pereira et al. reported the anodic stripping voltammetric determination of Se(IV) in standard certified solutions (Pereira et al., 2006) while Westbroek et al. employed a flow-through cell for the on-line determination of Ce(IV) (Westbroek et al., 2002). The latter setup was used to monitor the polymerization reaction of poly(ethylene oxide) (PEO) catalysed by cerium(IV) ammonium nitrate. Following the decrease in cerium ions concentration over time it was possible to obtain information on the polymerization kinetics and overall reaction rate.

3.1.2. Determination of small organic molecules

Since the beginning Au and Ag CD electrodes were employed also for the analysis of organic molecules, like pharmaceuticals, metabolites and others. . For example, Richter et al. (2004) fabricated twin gold microelectrode for amperometric detection in capillary electrophoresis; the flexibility of the design and the disposable nature of the double detector were successfully employed to detect simultaneously iodide, ascorbic acid, dipyrone, and acetaminophen. Patterning of the gold thin layer of CDs with different toner mask was used by Paixão and Bertotti to realize a disposable electronic tongue for milk adulterant analysis (Paixão and Bertotti, 2009).

A flow injection setup was coupled with Au CD electrodes for the determination of dipyrone in pharmaceutical formulation (Muñoz et al., 2001a) or combined with spectroelectrochemical detection for quantification of o-toluidine (Daniel and Gutz, 2001) and promethazine hydrochloride (Daniel and Gutz, 2003b). The combination of amperometric detection with absorptiometric signal was proved to be particularly useful to suppress the effect of other electroactive interfering compounds, without the need for complex sample pre-treatments. More recently, other pharmaceuticals have been determined thanks to disposable Au CD and DVD electrode, modified with different nanomaterials. Felix et al. modified Au CDtrodes with multi-walled carbon nanotubes for the quantification of terbutaline (Felix et al., 2016), while Daneshvar et al. employed a composite of copper nanoparticles and carbon nanotubes for the voltammetric detection of paracetamol (Daneshvar et al., 2016). Tarahomi et al. (2019) instead functionalized the surface of Au-DVD electrodes with graphene oxide/Ag nanoparticles/β-cyclodextrin composite for the quantification of naproxen in pharmaceutical formulation and urine samples.

Also the analysis of other relevant biological targets was addressed using disposable CDtrodes: ascorbic acid was quantified on platinum (Muñoz et al., 2001b) or manganese(III) (Leonardi et al., 2014) modified Au CD electrodes, while glucose was detected with an Ag-CD electrode modified with Ag nanoparticles (Kumachev et al., 2010) and a photoelectrochemical sensor based on Ag-DVD electrodes functionalized with VO_2/V_2O_5 (Zheng et al., 2016). Another nanomaterials-based sensor for the determination of para-nitrophenol was recently reported by Afzali et al. (2016). The surface of the Au DVD electrodes was modified with a nano-porous mercury/gold amalgam and para-nitrophenol was quantified by means of square wave voltammetry, also in several natural water samples.

A model analyte commonly used to test new analytical protocols is H_2O_2, however the quantification of hydrogen peroxide has also relevant real analytical applications (Halliwell et al., 2000; Stone and Yang, 2006). Several surface modification and fabrication protocols for Au and Ag CDtrodes were validated through the analysis of H_2O_2: the photochemical synthesis of Prussian Blue film on Au CDtrodes (Hu et al., 2005) was verified by amperometric analysis of hydrogen peroxide. Chen et al. reported an hydrogen peroxide sensor based on roughened Ag-CDtrodes (Lian et al., 2009). $_{22}$. Also Shafei and Honeychurch (2013) used an Ag-DVD electrode to quantify hydrogen peroxide in real water samples, attaining satisfying results in term of LOD (80 μM) and linear range (0.08 mM–3.5 mM). Another application of Ag-DVD electrodes for hydrogen peroxide determination was reported by Wen et al. (2013): they deposited a dendritic silver nanostructure on the electrode, through an electrochemical method of multi-potential step-scans. Ngamaroonchote et al. (2017) compared the performance of Au electrodes obtained from different compact disc supports. Compact Disc-Recordable (CD-R), Digital Versatile Disc-Recordable (DVD-R), Blue-ray Disc Recordable (BD-R), Compact Disc-Read Only Memory (CD-ROM), Digital Versatile Disc-Read Only Memory (DVD-ROM) and Blue-ray Disc--Read Only Memory (BD-ROM) were used to fabricate disposable electrodes and applied to the determination of H_2O_2. The authors evidenced sensible differences in the electrochemical response of electrodes based on different substrates and linked this effect to their different surface patterns, showed in Fig. 5. Indented pattern from ROM disc type offered better electrocatalytic signal compared with striped pattern from R disc type. The electrodes fabricated from BD-ROM, having the smallest scale of indented pattern (300 nm), showed the highest sensitivity towards H_2O_2 detection.

Silver DVD-R electrodes were also used as bipolar electrodes (BPE) for electrocatalysts screening and H_2O_2 sensing (Shayan and Kiani, 2015). With the obtained BPEs it is possible to follow visually the electrodissolution of the reflective layer at the anodic pole of electrodes. In this way is possible to obtain information about either the concentration of analyte in the solution or the electrocatalytic activity of the catalyst deposited on the cathodic pole of the BPE. The most effective electrocatalyst or the highest concentration of analyte results in oxidation of a longer length of the Ag DVD-R strip electrodes.

3.1.3. Biosensors application

The qualities and performances of CDtrodes opened up interesting possibilities for biosensing applications, thanks to their characteristics, comparable to traditional thin film electrodes (M. V. Foguel et al., 2016b). Conventional and well-studied functionalization protocols and biosensing architectures can also be employed on CDtrodes, with the added value of disposability and low cost. The highly ordered gold thin layer of CDs and DVDs was deemed ideal to build self-assembly monolayers (SAM) for bio recognition element immobilization: Li et al. (2008) studied DNA hybridization on inkjet-printed Au CDtrodes arrays, controlling the molecular orientation of thiolate DNA-SAM. Also for bio-probes immobilization different compact disc substrates gave different results, as reported by Foguel et al. (M. V. Foguel et al., 2016b). The authors compared the deposition characteristics of short oligopeptides on Au disposable electrodes obtained from three different CD-R, linking once again the performance of the CDtrodes to the nanostructured surface of the thin gold layer. DNA hybridization studies were also performed by Cheng et al. (2009) using Au CDtrodes to immobilize a methylene blue labelled probe able to capture the complementary DNA strand of a bacterial plant pathogen, *Ralstonia solanacearum*. The decrease in the mediator response upon binding with the target strand was used to identify the bacteria presence. Another biosensor, based on DNA hybridization recognition for single nucleotide polymorphism detection, was fabricated by Ahangar and Mehrgardi (2012) anodizing the Au electrode surface to obtain nanoporous gold; nanoporous electrodes enhanced the sensitivity of DNA biosensor and makes it capable of detecting complementary target DNA in sub-nanomole scales (ca. 30 pmol). Another application of DNA biosensing principles on CDtrodes was reported in 2013 by Frense et al.; they realized an impedimetric aptasensor for thrombin detection with an interdigitated Au-CD electrode, coupled with an homemade flow-through system (Frense et al., 2013). Continuous impedimetric detection of thrombin in buffer was achieved with the possibility to regenerate the aptamer *in-situ* by simple heat treatment.

Several immunosensors based on disposable CDtrodes were reported

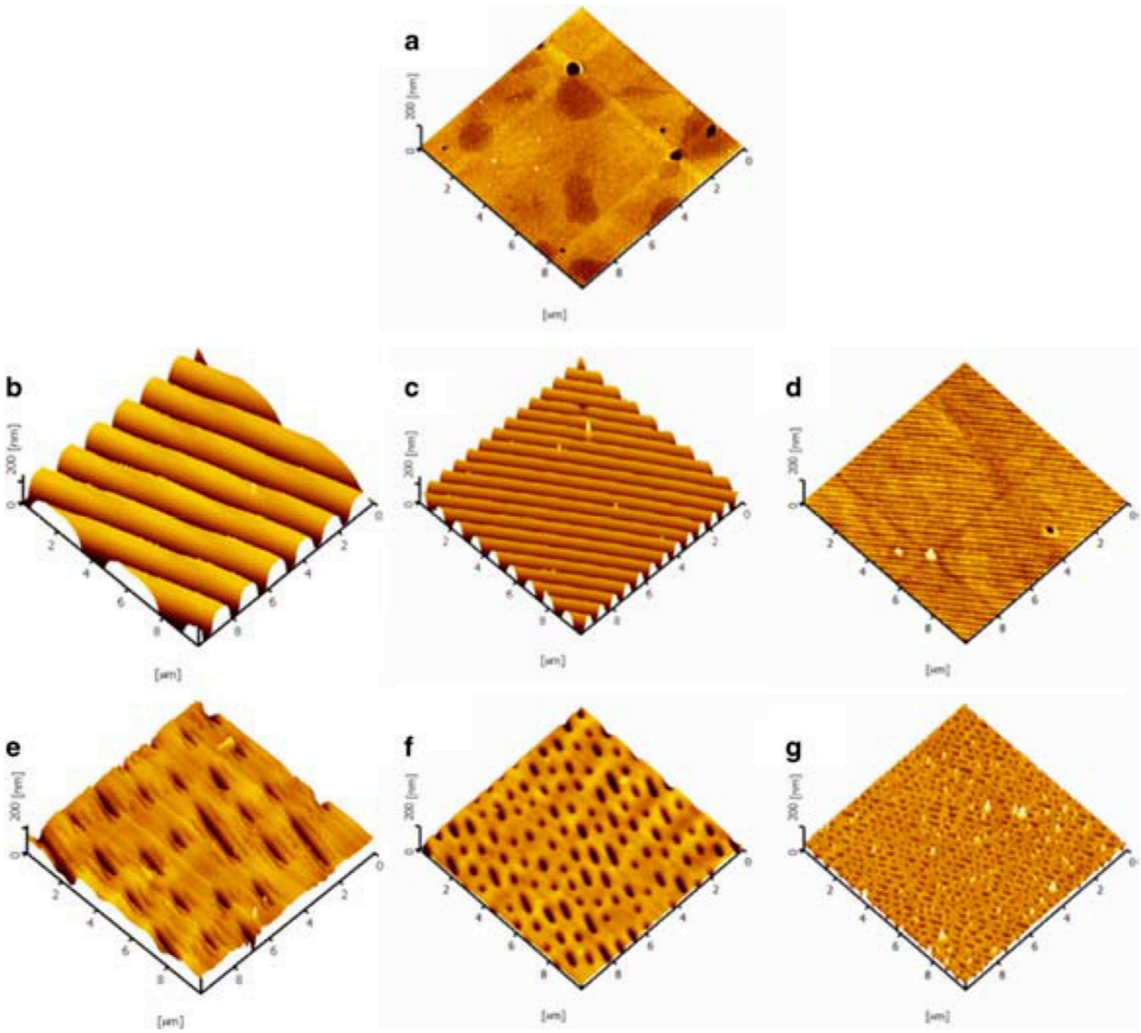

Fig. 5. AFM images ($10 \times 10\ \mu m^2$) of (a) gold film coated on a flat polycarbonate, (b) CD-R, (c) DVD-R, (d) BD-R, (e) CD ROM, (f) DVD-ROM and (g) BD-ROM; reported from (Ngamaroonchote et al., 2017).

in literature, aiming to obtain cheap and easy-to-produce screening tools for routine medical analyses also in developing countries: Au CD-R electrodes were used as a platform to develop an amperometric immunosensor for Chagas' Disease biomarkers (Vinicius Foguel et al., 2011) or for the impedimetric determination of dengue virus infections (Cavalcanti et al., 2012). The versatility of the fabrication processes to obtain CDtrodes was used moreover to build microarrays for cancer biomarkers detection in combination with microfluidics devices for point-of care diagnostics (Li et al., 2019; Tang et al., 2016, 2012). The surface of the CD was patterned by wet etching to obtain a network of microfluidic channels for sample handling and used for the impedimetric detection of the biomarkers at interdigitated electrode arrays, obtained from the same CD matrix. Also in the field of food and feed safety, the combination of Au disposable electrodes from compact disc with immunological biorecognition elements was used to build a low-cost label-free impedimetric biosensor for determination of aflatoxin B_1 in peanut samples (M. Foguel et al., 2016a).

Being the use of CD/DVD the first example in the history of waste based electrodes, it is important to report also on the findings and the protocols developed, which can be applied to other waste materials. The research done to understand the surface features, the production processes and the best configuration for diverse analytical application might still be of paramount importance in the development of the new waste based disposable sensors.

3.2. Other recycled materials

Many other waste materials can be utilized for the fabrication of disposable electrodes or surface modifiers, starting from e-waste like printed circuit boards. In the early 2000', Shih et al. reported the realization of a copper modified SPE for amperometric detection of arbutin (Shih et al., 2005) and ascorbyl glucoside (Shih and Zen, 2006): the copper was recovered from printed circuit board wastes. et al.Looking at more recent electroanalytical applications, Honeychurch published a report on determination of lead with carbon rod electrodes obtained from spent zinc–carbon batteries (Honeychurch, 2019). The obtained electrodes showed performances comparable to traditional carbon rod electrode also for lead determination in tap water. Graphitic materials are the most commonly obtained from different kind of waste, both organic and inorganic. For example, Randviir et al. fabricated carbon paste electrodes with hydrothermally treated textile wastes (Randviir et al., 2019). Varying the digestion treatment conditions, the authors managed to obtain carbon paste electrodes made 100% from textile waste hydrochars with good conductivity even if rather poor reproducibility. Also highly porous carbon ($1625\ m^2\ g^{-1}$) was obtained from waste tires and used in a composite polyaniline based supercapacitor (Boota et al., 2015). As already mentioned electrodic metals can also be recovered from waste materials, like copper from electric cables (Abdelbasir et al., 2018). The authors modified graphene electrodes with nanocuprous oxide obtained from waste electric cable and applied

the modified electrode to the detection of dopamine.

The combination of the approaches mentioned in this section, for environmentally friendly electroanalysis, is indeed gathering attention and a few examples of platforms completely obtained from waste materials started to appear. One very recent example, showed in Fig. 6, reported the fabrication of SPE on repurposed PET (polyethylene terephthalate) soft drinks bottle using a DIY ink composed of graphite pencil powder and nail polish (de Araujo Andreotti et al., 2019). The obtained disposable SPEs were used for the determination of hydroquinone (HQ), epinephrine (EP) and serotonin (5-HT), also in real samples.

Several reports have been published recently on the recovery of indium-tin oxide (ITO) electrodes from commonly used electronic displays: ITO have been obtained from photovoltaic devices (OPVs) (Dang et al., 2015) or organic light-emitting diodes (OLEDs) (Dang et al., 2017). Liquid crystal displays (LCDs) have been also used as substrate for the fabrication of thin film graphene electrodes (Divyapriya et al., 2018). Given the ubiquitous nature of these displays technologies (laptop, smartphones, TVs) these last example shows once again that is possible and indeed necessary to find novel ways to re-use electrodic materials from e-waste, and, as shown in this section for the CD/DVD electrodes, electrochemical applications are and will be at the forefront in this regard.

4. Green nanomaterials and e-waste metals recovery

Along with recycling and reusing waste materials for electroanalysis, in recent years different research groups have been trying a greener route to the synthesis of electrode modifiers or the realization of disposable electrodes, obtained from vegetable matter (Samaddar et al., 2018). Regarding graphitic carbon materials, several examples have been reported for energy storage applications. Jiang et al. (2014) converted bamboo chopsticks in uniform carbon micro fibres using a simple delignification hydrothermal process in alkaline solution. Another example from Xu et al. (2016) dealt with the conversion of crop stalks in two-dimensional hierarchical porous carbon. Again after a green graphitization process based on metal salts ($FeCl_3$ and $ZnCl_2$), the obtained 2D carbon sheets were loaded with TiO_2 and $LiMnPO_4$ nanoparticles and used as cathode and anode in a lithium-ion battery. Wang et al. reported a simple wet spinning method to obtain conductive carbon nanofibers based on cellulose/lignin composite (Wang et al., 2019). The fibres show a high conductivity and good mechanical stability compared to other kind of cellulose-based carbon fibres. While all the above mentioned materials are used in energy storage applications, they present many sought-after characteristics for (bio)electroanalytical applications, such as large surface area or the possibility to graft/adsorb biomacromolecules.

Even if only one example (Bastos-Arrieta et al., 2018), at the best of our knowledge, have been reported up to now in the application of green electrode modifiers to screen printed electrodes specifically, several eco-friendly routes to the synthesis of electrocatalytic nanomaterials are present in literature (Mittal et al., 2013). Synthesis of noble metals nanoparticles based on reducing and capping agents obtained from renewable plants sources seems a promising approach to more environmentally benign synthesis: silver nanoparticles (AgNPs) were obtained from avocado pit (Lynk et al., 2018), bilberry and red currant (Zuorro et al., 2019), grape stalk extracts (Bastos-Arrieta et al., 2018; Carbone et al., 2019) or from food wastes (Sivakumar et al., 2013; Xu et al., 2014). Also gold nanoparticles (AuNPs) can be synthesized using natural by-products like eggshells (Devi et al., 2012), grape stalks (Krishnaswamy et al., 2014) or plums extract (Dauthal and Mukhopadhyay, 2012). Even if Au and Ag are the most commonly employed metals, also other examples of nano-objects obtained with green synthetic routes have been reported. A wide variety of possible electrode modifiers could be obtained from renewable sources and waste materials; for example, nanoparticles based on Pd (Eroglu et al., 2013; Lakshmipathy et al., 2015; Parker et al., 2015), magnetic Fe_3O_4 (Lunge et al., 2014; Mahdavi et al., 2013; Venkateswarlu et al., 2013), TiO_2 (Bao et al., 2013), ZnO (Azizi et al., 2014) and Mn_3O_4 (Yan et al., 2014). All this nanomaterials can be easily integrated with screen printed and disposable electrodes, and their use for improving the performances of sensors and biosensors was extensively reported (Lv et al., 2018; Maduraiveeran et al., 2018).

Regarding nano-carbon modifiers, highly graphitized carbon nanostructure and carbon nanotubes (CNTs) can be obtained from a variety of renewable sources. While CNTs, both single walled and multi walled are obtained mainly from vegetable oils, graphene-like nanostructure have been synthesized from an assortment of different carbon sources like food wastes and agricultural or husbandry by-products (see Table 1 for an overview).

All the above mentioned examples show the possibility to reuse agricultural by-products to obtain electrocatalytic electrodes modifiers with high added value and simple synthetic protocols. In the broader context of eco-friendly analytical applications of SPE, these modifiers may represent a good alternative to more traditional approach to electrode functionalization.

If on one side, sustainability asks to redesign new production processes starting from renewable sources, on the other side it requires to recycle and recover precious, nonrenewable materials. For this reason, in this paragraph it is important to mention also the numerous protocols currently used to recover various metals (lithium, nickel, cobalt,

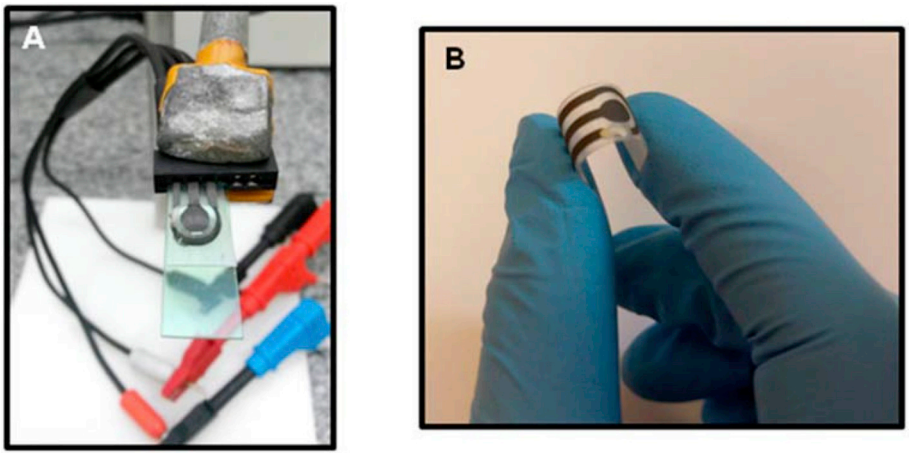

Fig. 6. Example of SPE made from soft drink plastic bottles, reported from (de Araujo Andreotti et al., 2019): (A) in-house built electrode holder with connections and the design of polyethylene terephthalate screen-printed electrode (PET-SPE); (B) folded PET-SPE, demonstrating the ink flexibility.

Table 1
Carbon sources for the green synthesis of nano-carbons, adapted from (Kumar et al., 2016).

	Source	Ref.
Carbon nanostructure	Camphor	(Kalita et al., 2011, 2010; Ravani et al., 2013; Sharma et al., 2013)
	Tea-tree extract	Jacob et al. (2015)
	Foods (cookie and chocolate), waste (grass, plastic, dog faeces) and insect carapaces	Ruan et al. (2011)
	Vegetation wastes (wood, leaf, and fruit), animal wastes (bone and cow dung)	Akhavan et al. (2014)
	Honey, sugar, butter and milk	Seo et al. (2013)
	Cheese	Seo et al. (2015)
	Camphor leaves	Shams et al. (2015)
	Sugarcane waste	Somanathan et al. (2015)
	Rice husk	Muramatsu et al. (2014)
	Wheat straw	Chen et al. (2016)
	Mango peel	Shah et al. (2018)
	Palm-based waste cooking oil	Azam et al. (2018)
MWCNTs	Camphor	(Antunes et al., 2011; Kumar et al., 2004; Kumar and Ando, 2005, 2003a; 2003b, 2003c; Somani et al., 2009)
	Turpentine oil	(Afre et al., 2006, 2005; Awasthi et al., 2010)
	Neem oil	(Kumar et al., (2011a))
	Castor oil	Awasthi et al. (2011)
SWCNTs	Camphor	(Kumar and Ando, 2005, 2003a; 2003c)
	Turpentine oil	Ghosh et al. (2008)
	Eucalyptus oil	Ghosh et al. (2007)
	Palm oil	Suriani et al. (2009)
Doped CNTs	Camphor	Ghosh et al. (2009)
	Turpentine oil	Ghosh et al. (2009)
	Sunflower	Kumar et al. (2011)
	Chicken feather	Gao et al. (2014)

manganese, zinc, copper, etc.) (Heydarian et al., 2018; Zhang et al., 2019) as well as precious ones, particularly gold and silver (Natarajan and Ting, 2015). These e-waste treatments allow to extract the metals by separating them from the other non-metallic components of circuits and sensors, such as resins, ceramics, paper, plastics, etc. (Marra et al., 2018; Naseri et al., 2019). They are mostly based on mechanochemical (Zhang et al., 2017), bioleaching processes (Natarajan and Ting, 2015) or hybrid technologies (Sahni et al., 2016). In bioleaching, microorganisms are used to solubilize and recover the metals following different paths. The increasing interest in this kind of bioprocesses is related to their selectivity towards (1) metals, (2) ease of operation, (3) cost-efficiency and (4) low environmental impact (4). The possibility to carry out bioleaching with different groups of microorganisms (i.e. chemolithotrophic prokaryotes, heterotrophic bacteria, fungi) makes these approaches even more versatile (Işıldar et al., 2019). Among the metals used in disposable electrodes, gold was the most investigated in recovery and bioleaching studies. Liu et al. proposed a comparative study of gold bioleaching with *Chromobacterium violaceum* strain, using different protocols (i. e. the one-step, the two-steps and the spent medium leaching protocol) (Liu et al., 2016). Although the leaching efficiency was not easy to estimate, the authors clearly described the potentialities of this biohydrometallurgic technology. Recently, Baniasadi et al. (2019), described the advances in bioleaching of gold, stressing the need of an in-depth understanding of the processes kinetics for a wider application of this technology. According to the authors, a further enhancement of the bioleaching capabilities should be achieve also by applying genetic makeup and generate microorganisms that are more efficient. Although the numerous open questions, these applications showed promising results with gold recovery percentage of 30–70% (Liu et al., 2016; Natarajan and Ting, 2015). The possibility to apply these treatments also to spent disposable electrodes seems a feasible alternative.

Such recovered metals can be used as components for conductive inks, or in a circular economy perspective, as precursor for the realization of green nanomaterials, thus combining eco-friendly synthetic routes with recovered and recycled e-waste.

5. Examples of SPE recycling

Despite the disposable nature of SPEs, the possibility to reuse them multiple times even for different purposes was always a topic of interest. For this reason, the maximum extent of reuse, the longevity and the effect of reactivation protocols (to be applied after-use) was investigated particularly on carbon-based SPEs. Churinsky et al. (Churinsky and Grgicak, 2014) tested repeated activation procedures using Tris-HCl buffer and PBS on commercially available carbon SPE cards. A relevant loss in the SPEs performances was observed when the WE of the reused SPEs undergo an electrochemical re-activation treatment. These studies first underlined the difficulties in cleaning and regenerating the electrodes surface of commercial SPEs after the first use. The possibility to use SPEs as precursor materials for the production of nanoparticles (Agrisuelas et al., 2017) suggested other ways to partially recycle (and not simply reuse) these devices. In this last work, the platinum was extracted from waste SPEs using aqua regia and then electropolymerized in the form of nanoparticles at the surface of a new SPE, obtaining an hydrogen peroxide sensor. The selective extraction of silver and platinum can be operated simultaneously through an acid treatment. Apart for the synthesis of hybridized nanoparticles (Gómez-Monedero et al., 2019), the metal solutions obtained can be used for the study of fundamental electrochemical processes in didactic laboratories (González-Sánchez et al., 2018).

Queiros et al. (Queirós et al., 2013) showed how to reuse SPEs without any pretreatment or cleaning steps. They used the silver contacts of spent commercial SPEs to realize a potentiometric sensor based on molecularly imprinted polymers and MWCNTs for the detection of bacterial toxins (see Fig. 7). Despite the good results presented in the mentioned studies, there are still very few examples of recycling paths for screen printed electrodes.

6. Conclusions and future perspectives

In the broader context of green electronics, the present review provides an overview of the advances in the production of renewable, recyclable and recycled disposable electrodes. Currently, research is ongoing to develop electrodes based on paper or cellulose materials as

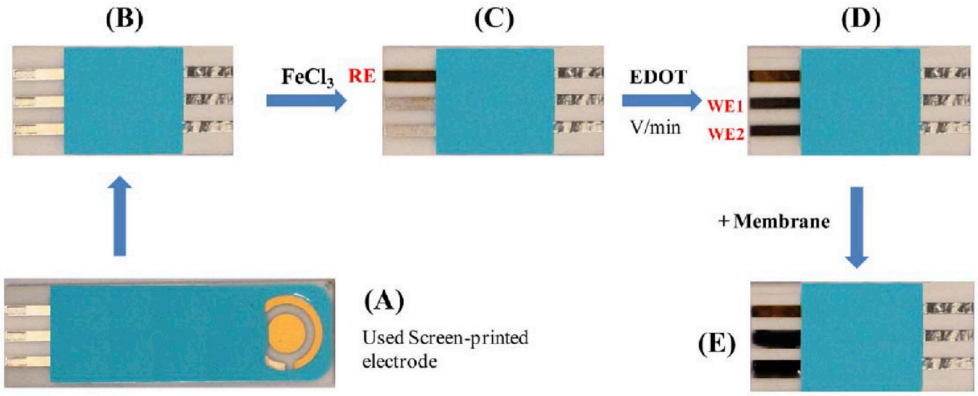

Fig. 7. Biparametric disposable chip construction reported from (Queirós et al., 2013). The ceramic support of screen-printed electrodes (A) with three silver contacts was cut to remove the indicating, reference and counter electrode support material. The insulating polymer on top was removed to expose the conductive silver (B). One of the sides of the ceramic was used as RE and WE while the other side was used as electrical contact. A $FeCl_3{\cdot}6H_2O$ solution was deposited on the silver track acting as RE, to allow the formation of a silver chloride on top of it (C). Each WE was first modified with a poly (EDOT) layer. The deposited polymer was then reduced at −0.5 V for 60 s (D). The ceramic support was then let dry and the selective membranes casted on top of each WE track (E).

well as recycled materials, such as PET bottles. Additionally, research on the retrieval of resources from discarded CD/DVD and green materials has been reported. Notwithstanding the urgent need to design sustainable analytical sensors, preventing e-waste growth and concurrently fostering the rapid development of new materials, the fabrication of biodegradable or recyclable electrodes still presents many open challenges. From reproducibility to safety and reliability to cost-effectiveness and scalability, the mass production of environmentally friendly and completely recyclable sensors and biosensors should be posed as one of the main goals of the electro-analytical chemistry community for the years to come.

Current possibilities are focusing on the implementation of either recycled or renewable materials for the substrates as well as for the electrodes themselves. The ultimate redesign goal is to grow towards a fully circular system, where re-use is mostly preferred. In a long term horizon, the development of completely biological sensors based on conductive microorganism such as cable bacteria (Meysman et al., 2019) could be envisioned, paving the road to biodegradable electronics or implantable diagnostic and therapeutic devices.

An issue that still need to be addressed is the risk of contamination. Most SPE and commonly used surface modifiers for biosensing are currently made with materials that may pose a threat to human health and environment. For example carbon nanotubes may be linked to increased risk of pulmonary inflammation, fibrosis, and granuloma in lungs (Narei et al., 2018); also the effects of metal nanoparticles on living organism are far from be fully elucidated (Buchman et al., 2019). Therefore, methods to safely dispose SPEs as non-hazardous waste are needed along with effective strategies to re-use and recycle them. Recycling strategies along with efficient waste disposal must be integral part of this new outlook on sensors and biosensor development.

Given the outstanding results obtained in the field of green electronics in the last decade (Irimia-Vladu et al., 2012; R. Li et al., 2018a) the possibility to redesign also the world of disposable electrodes is truly at hand and more efforts should be devoted to this objective, combining different expertise and cross-pollinating diverse analytical fields with increased awareness on environmental issues.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Giulia Moro: Conceptualization, Investigation, Writing - original draft. **Fabio Bottari:** Conceptualization, Investigation, Writing - original draft. **Joren Van Loon:** Writing - review & editing. **Els Du Bois:** Writing - review & editing. **Karolien De Wael:** Supervision, Writing - review & editing. **Ligia Maria Moretto:** Supervision, Writing - review & editing.

Acknowledgments:

This research received funding from FWO and IOF (UAntwerpen).

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