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3D printing in chemical engineering: A review

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Abstract

3D printing, also known as additive manufacturing has become one of the most revolutionary and powerful tools serving as a technology of precise manufacturing of chemicals ranging from laboratory scale to large scale production. There is constant motivation towards designing new concepts of manufacturing with high efficiency. The introduction of 3D printing technology in the chemical industry has opened new horizons in the research and development of printed materials and equipment. One of the fields of technology, art, and science that is currently advancing the fastest is three-dimensional printing, and its uses are continually expanding. Three important elements play a big role in the rapidly expanding usage of 3D printing. First, as a result of lower raw material costs, increased competitive pressure, and technological developments, 3D printing is becoming increasingly affordable. Second, the rate at which materials can be printed is getting faster. Third, more types of materials can now be used with new 3D printers. A wide variety of polymers, resins, plasticizers, and other materials are being employed to make novel 3D products as a result of advancements in the chemical industry. In this review, we discuss the contribution of three-dimensional printing in the field of chemical engineering.

Keywords: Microfluidics; Additive manufacturing; Fused deposition modelling; Electrodes; Active pharmaceutical ingredient

1 Introduction

The field of 3D printing is constantly evolving in both academic and industrial research environments. The development of 3D printing technologies has opened up new possibilities for implementation in the field of rapid prototyping, instrumentation, dentistry, microfluidics, biomedical devices, tissue engineering, drug delivery, etc. Thanks to the huge reduction in costs and common commercial availability, 3D printing has become a cutting-edge technology with huge potential - also for teaching and applied chemistry. It opens up the possibility of printing custom-made reactors such as (micro) flow reactors. In addition, 3D printing technology can simplify chemical reactions such as heterogeneous catalysis, as reactants such as catalyst can be immobilized in the reactor by direct printing. Thus, chemical experiments can be printed and it is possible to quickly transform an idea into a process or a concept into an educational experiment as an elegant think-and-print approach [1].

The chemical industry will be both a provider of the new generation materials needed for 3D printing and a beneficiary of this process. The chemical sector has a huge opportunity to create unique consumables and generate new revenue streams with 3D printing. Using this technology, the concept is converted into a prototype using computer-aided design (CAD) files, enabling the production of digitally controlled, customized products. In this technology, layers of materials such as living cells, wood, alloy, plastic, metal, etc. are stacked on top of each other to form the required 3D object. Chemical synthesis is usually carried out in laboratories using expensive and complex equipment, which often hinders research progress. It is now viable to use 3D printing to produce reliable and durable miniature fluidic reactors as

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"micro-platforms" for organic chemical synthesis and material processes that can be produced in a short time using inexpensive materials. Such microreactors enable multi-step synthesis of target molecules and identification of individual parts using reagents that could be incorporated during the 3D printing process.

Some chemical companies use 3D printers to maintain process plant assets in addition to printing lab equipment. For example, if an asset fails due to a broken engine valve, a replacement part can be printed locally and installed immediately. On-site manufacturing of spare parts can significantly reduce inventory costs, thereby increasing overall equipment uptime. There is a network of on-demand manufacturers who can print and supply parts on demand for businesses that don't want to make the parts themselves. All industries can benefit from 3D printing's promise to lower supply chain costs. For example, by increasing equipment uptime and streamlining work management, on-demand spare parts printing capacity can save costs. With less waste and less carbon impact, 3D printing also helps control costs. 3D printing is an additive process that uses only the required amount of material, unlike conventional "subtractive" manufacturing methods that remove raw materials from the product. In this way, significant savings in raw materials can be achieved.

2 Applications in Chemical Engineering

2.1 Microfluidics

Microfluidics is the science and technology used in channels with a range of 10-100 micrometres to control a small amount of fluid. 3D printing has a major impact on the field of microfluidics and lab-on-a-chip technology. While the possible functions of 3D printed microfluidic devices are far-reaching, the use of 3D printing for bioanalytical research appears to be a likely extension of previous efforts made in 3D environments to control cell patterning using soft lithography [2]. The fabrication of a complex microvascular network composed of 100–300 micrometre cylindrical channels capable of diffusion-based mixing under laminar flow profiles as well as mixing from turbulent flow is one of the first examples of 3D printing for microfluidic applications [3]. Cronin was among the first to recognize the potential of additive manufacturing in preparative chemistry with his "reaction ware", which uses fused deposition modelling (FDM) techniques to produce chemical reactors [4]. Using stereolithography, an electrochemical flow cell was fabricated that can be integrated with electrodes without the use of adhesives. To characterize the mass transport in the flow cell, the oxidation of ferrocenylmethyl trimethylammonium hexafluorophosphate was monitored using a two-electrode arrangement with a working electrode made of either gold or a diamond strip doped with polycrystalline boron and a quasi-reference electrode of silver wire coated with silver chloride. Such a device has a potential impact on future analytical and kinetic flow measurements [5].

Currently, stereolithography is the most promising approach for the routine creation of microfluidic structures, but several approaches in development also have potential. Microfluidic 3D printing is still in its infancy, much like PDMS was two decades ago. With further work on improving the hardware and software control of the printer, expanding and improving the selection of resins and printing materials, and realizing other applications for 3D printed devices, we anticipate that 3D printing will become the dominant microfluidic manufacturing method. Microfluidic devices made of hard non-cytotoxic materials such as silicon and glass have many advantages for the analysis or actuation of biological samples. Chips can be produced by standard cleanroom processes. The channel geometry provides a closed and therefore controlled environment that can be optimized for the sample [6]. Although 3D printed microfluidics is developing rapidly, some challenges still remain. The limitations of 3D printed microfluidics can be divided into two groups, accuracy and material. There is a certain distance in accuracy between 3D printing and traditional microelectromechanical systems (MEMS) technology, which means that most 3D printing methods are not suitable for the production of nanofluids. Leakage can be a problem when D3DPs are used to directly print microfluidics. Furthermore, although a 3D printer is an efficient and convenient tool for microfluidic fabrication, it does not mean that a 3D printer can print everything [7].

Table 1 Comparison of each printing method [8]

Method	Principle	Material	Advantages	Disadvantages	Suitable Microfluidics
FDM	Extrusion-based	Thermoplastic, eutectic metal, ceramics, edible material, etc	Simple using and maintaining, low cost, easily accessible	Rough surface, low resolution	Mould casting, channel size larger than 200 mm, Low-cost chips
SLA & DLP	Photocuring	Liquid photosensitive resin	High accuracy	Limited resin, unbio-compatible	Mould casting, Channel size larger than 100 mm
3DP-LR	Inkjet-based	Liquid photosensitive resin	High accuracy	Very expensive	Transparent chips
SLS & SLM	Photo melting	Powdered plastic, metal, ceramic, PC, acrylic styrene, PVC, ABS wax, etc	Wide adaptation of materials, high accuracy, high strength	Very expensive	Reactor with high temperature
LOM	Paper cutting	Sheet material (paper, plastic film, metal sheets, cellulose etc.	Low cost, easy to manufacture large parts	Time-consuming, low material utilization	3D micro PADs with different agents
3DP-P	Inkjet-based	Powdered plaster, ceramics sugar etc.	Colourful printing	Post surface treatment, low strength	Unsuitable
LDW & Two-Photon Polymerization Process	Laser-based	Glass, fused silica etc.	High accuracy	Expensive	Situations need high accuracy

2.2 Electrochemistry

Electrochemistry is another branch of science that can certainly benefit from 3D printing technologies, paving the way for the design and production of cheaper, more powerful and ubiquitous electrochemical devices. 3D printing can be used to produce conductive electrodes with unique shapes or compositions for use in redox and catalytic processes, as well as the construction of fluid handling systems such as voltammetric cells or subsequently integrated with the electrodes of micro-macrofluidic systems. Currently, not every 3D printing process for manufacturing electrodes for energy applications is described in the literature. Because electrodes and electrode support must be conductive, 3D printing techniques that use insulating precursor materials require additional processing steps, such as electroless deposition or puttering, to introduce conductive materials to the electrode surface. Thus, the most commonly reported 3D printing processes for the production of electrodes for electrochemical energy applications are FDM using carbon fibres, DIW using conductive inks, and SLM using metal powders. Recently, a complete electrochemical flow cell was fabricated by additive manufacturing using FDM printing and then tested for mass transport and electrochemical experiments. Nickel foil electrodes have been integrated and assembled in a printed cell device in addition to a liquid handling system [9].

2.2.1 Electrodes

Electrodes are the foundation of the battery's structure and performance. 3D printing of electrodes and devices enables various properties related to geometry, stiffness, porosity and size. The choice of precursor materials and the type of 3D printing techniques allows control over these properties. The size of parts that are 3D printed can vary in size from the millimetre scale to the meter scale, again depending on the 3D printing technique [10],[11]. In the past, more attention was paid to the research of advanced electrode/electrolyte materials rather than to the structural design, manufacturing process and assembly of the electrodes/electrolytes. Well-designed structural electrodes/electrolytes are the bridge to transform advanced energy materials into high-performance devices.

Recently, 3D structural electrodes/electrolytes with porous microstructure have been strongly promoted for higher power/energy density and better safety [12]. 3D structural electrodes/electrolytes could provide shorter diffusion paths and larger available interfacial surface areas than conventional 2D planar electrodes/electrolytes. Therefore, 3D electrode/electrolyte design and fabrication are becoming more and more crucial in battery systems. Several researches and reviews have also reported and summarized the 3D structures of electrodes and electrolytes [13]– [15]. Activated carbon, carbon black (amorphous carbon), and graphite are widely used materials for the preparation of conductive 3D printable compositions due to their electrical conductivity, low cost, ease of handling and production, chemical and electrochemical stability, and high porosity (especially for activated carbons). Some of these materials are also capable of reversibly intercalating Li ions and have significant intrinsic specific capacity [16], [17]. They have been used in various 3D printing techniques (extrusion, inkjet printing (IJP); selective laser sintering (SLS)) in the form of conductive polymer-based composites used for the production of electrically conductive structures, supercapacitor electrodes, Li-ion battery electrodes [18]– [20].

Energy storage devices such as batteries or supercapacitors play an important role in modern society, and the main goal of energy storage device development is to achieve the requirement of high energy density and high-power density while maintaining long cycle life under practical operating conditions [21]. Among these components of energy storage devices, electrodes play the most important role in deciding their electrochemical performance. Additive manufacturing technique makes the advanced electrode architecture design possible and focuses on improving the performance of energy storage devices [22].

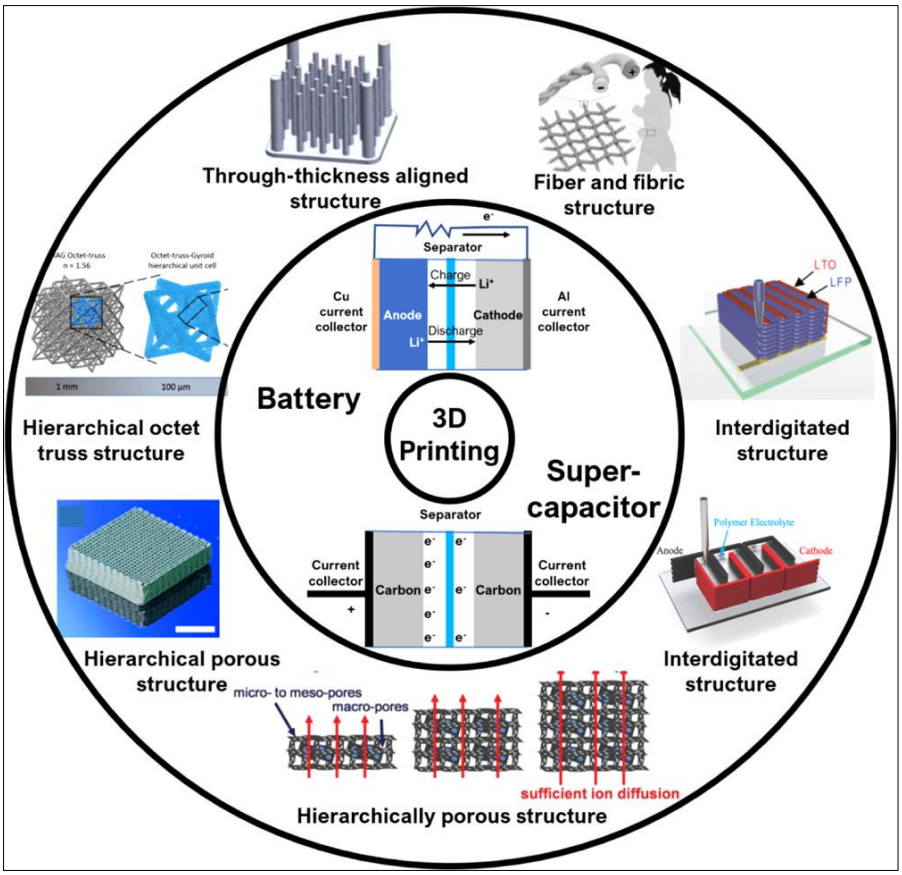

Figure 1 Overview of three-dimensional (3D) printing for emerging advanced electrode architectures. [23]

2.3 Metal 3D printed electrodes

Metal 3D printing provides an easy and fast way to produce electrodes. However, in relation to water splitting, currently available precursor metal powders for 3D printing (e.g. Ti, Cu, stainless steel and Al) are not known to be effective; therefore, it is imperative that metal-printed electrodes be modified to compete with current state-of-the-art water splitting catalysts [24]. Electrodeposition is most widely used for depositing active materials for 3D printed electrodes. Any electrode size or shape can be modified by electrodeposition with a large number of active water-splitting materials such as IrO2, Pt, Ni, NiP, NiFe oxide, MoS2, and Ni-MoS2 [10]. Selective laser melting (SLM) coupled with anodization

and Atomic layer deposition (ALD) has previously been used to tailor the surfaces of 3D printed metal electrodes for PEC water oxidation [25],[26]. In one study, SLM was used to create stainless steel electrodes with a square geometry; due to the non-existent PEC water oxidation properties of stainless steel, the surfaces of the 3D printed electrodes were modified with TiO2 by ALD [25]. The ALD process was performed using TiCl4 as precursor and H2O as reactant. The thickness of the TiO2 layer deposited on the 3D-printed electrode depended on the number of preformed cycles. In this study, three TiO2 layers of different thicknesses were deposited and tested as PEC water oxidation catalysts. Prior to electrochemical evaluation, TiO2 layer thicknesses were investigated by ellipsometry and were determined to be 28, 54, and 77 nm for ALD cycle numbers of 400, 800, and 1200. PEC water oxidation studies showed that performance increased with increasing TiO2 thickness, which was related to an increase in layer crystallinity [25]. Special attention has been paid to 3D printed carbon electrodes due to the high electron transfer of neurotransmitters on carbon surfaces facilitated by the adsorption step. However, carbon electrodes may not be completely selective for the detection of one species in different biological media, because other molecules also undergo electrochemical oxidation processes on the same surface, for example ascorbic acid, dopamine, serotonin, epinephrine, noradrenaline are present in the brain slice [27].

3. Pharmaceutical Industry

In 1989, Scott Crump filed a patent on fused deposition modelling. Using this technique, the object is formed by depositing layers of solidifying materials (self-hardening waxes, thermoplastic resins, and molten metals) until the desired shape is formed [28], [29]. Spritam®, the first 3D-printed prescription drug product, received FDA approval in 2015 to treat partial onset seizures, myoclonic seizures, and primary generalized tonic-clonic seizures. Since then, many innovations have been evolved using the 3DP technology.

The growing demand for customized pharmaceutical and medical devices means that the influence of additive manufacturing has increased rapidly in recent years. 3D printing has become one of the most revolutionary and powerful tools serving as a technology for precision manufacturing of individually developed drug forms, tissue engineering and disease modelling. Current achievements include multifunctional drug delivery systems with accelerated release characteristics, adjustable and personalized dosage forms and phantoms matching the specific anatomy of the patient, as well as cell-based materials for regenerative medicine. Hot melt extrusion (HME) as well as semi-solid extrusion are well-established processes in pharmaceutical technology. The growing popularity of printing methods based on this technical solution is related to the progressive availability of compact dimensions and relatively inexpensive equipment [30].

In principle, two printing methods can be distinguished:

- Extrusion of semi-solid or semi-melted materials (gels, pastes) at room or elevated temperature.
- Extrusion of molten thermoplastic rod-like material (filament).

Thanks to the possibility of using different materials, 3D printing methods have a wide application in medicine, for example to build spatial systems used in tissue engineering as well as in pharmacy to prepare such drug forms as tablets, capsules or implants [31].

3D printing makes it possible to individualize the drug according to the patient's body weight and lifestyle by modifying the dosage form, e.g. orodispersible tablets instead of conventional tablets for active or non-compliant patients. The simplicity of the preparation of drugs with different doses is due to the scalability of the designed objects, so that the dose can be controlled by the calculated material consumption when changing the size of the printed object already at the design stage. This manufacturing method appears to be particularly advantageous in the manufacture of orphan drugs manufactured for small groups of patients. The relatively low cost of producing dosage forms with different dosages is one of the main advantages in terms of short batches of the medicinal product [32], [33]. Rapid production of single batches could be achieved in time or resource limited environments; for example, within hospital trauma and emergency and acute medical care units, disaster areas, ambulances, military bases and in low- and middle-income countries [31].

3D printing gives the possibility to produce tablets with more than one active substance characterized by different properties and different dissolution profiles. It can therefore lead to a reduction in the number of used preparations of complex drug formulations [32]. By using 3D printing technology, precise control of the dissolution behavior can be achieved mainly by applying selected soluble or insoluble excipients, but also by designing the specified geometry and internal structure of the printed drug forms [34]. However, this option should only be used by healthcare professionals,

as it requires knowledge of the pharmacokinetics of the medicinal substance and the patient's health status, and it can be applied in hospital pharmacies.

The introduction of additive manufacturing in the clinic reduces the time and cost of medical treatment and improves the success of operations. This has led to the development of new surgical procedures, especially high-risk ones that are rarely performed. In addition, 3D printing of highly mimetic organ models for surgical training can facilitate and shorten surgery time and reduce intraoperative complications [30]. 3D printing can easily be introduced into pharmaceutical composition in pharmacies. Fused deposition modelling appears to be the closest route, as with a good quality active pharmaceutical ingredient (API)-loaded filament and a database of printable objects, a medically trained staff, i.e., a pharmacist, can print the final dosage form with a defined architecture and dose of active ingredient [35]. The production of API filled filaments is not a big challenge for the pharmaceutical industry, as hot melt extrusion, which is the basic method of filament preparation, is well established in pharmaceutical production. One of the advantages of this 3D printing method is the ability to produce filaments with an embedded API in a crystalline state, which can be disordered during 3D printing. This approach can overcome the problem of stability of amorphous drugs that are often formed during 3D printing due to the mechanism of the 3D printing process, which is often based on melting the API with the polymer or rapid evaporation of the solvent from the drug solution [36].

In addition to immediate or sustained drug release, 3DP technology is applicable to other types of controlled release tablets. Using three different grades of Hypromellose acetate succinate (grades LG, MG and HG), enteric tablets were manufactured by FDM to produce delayed-release tablets without the need for an external enteric coating [37].

Other 3DP methods such as SLA and IJ method are available for tablet production. For example, SLA was successful in producing ER tablets of 4-aminosalicylic acid or paracetamol using polyethylene glycol diacrylate, diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide, and polyethylene glycol in different formulation ratios where the drug release profiles varied. Depending on the composition of the formulation [38].

To combine complex treatment regimens into one, multiple APIs can be loaded into a single tablet, called a polypill. In recent studies, 3DP technology has been used to produce polypills exhibiting controlled release profiles [32], [39]. To use 3DP to control more complex release profiles, different shapes of drug carrier templates (or moulds) are produced. Through complex templates, it is possible to create tablets that contain multiple components, generating a multi-active release profile. In this way, APIs are not only released in zero or first order, but more complex release profiles can be obtained.

While the possibilities of 3D printing are still being explored, the integration of 3D printing into industry will require a shift in the business model and approaches will need to be carefully considered before 3D printing takes on pharma on a large scale.

While the evidence base for 3D printing in pharma is extensive, more work needs to be done for all parties to have confidence in the technology. Several regulatory and technical challenges remain, including strategies to ensure the quality and safety of manufactured drugs. Manufacturing conventional drugs requires GMP compliance and extensive testing requirements, including cross-contamination risk assessment, weight uniformity, and stringent cleaning requirements [40].

Despite the fact that 3DP has been around since the 1980s, there is still a lot of research being done in this area, particularly in regards to the development of materials that are appropriate for use in pharmaceutical and medical applications. The 3DP of new, adaptable materials with the capacity to change their characteristics when subjected to external influences or over time is one of the ongoing studies in the field. The structural modification over time or otherwise called the fourth dimension, created a new term called “4D printing” [41]. In oral dosage forms, this technology allows the modification of drug delivery, since the timely release profile can be triggered by stimuli, such as pH, temperature, enzymes action, and time [42].

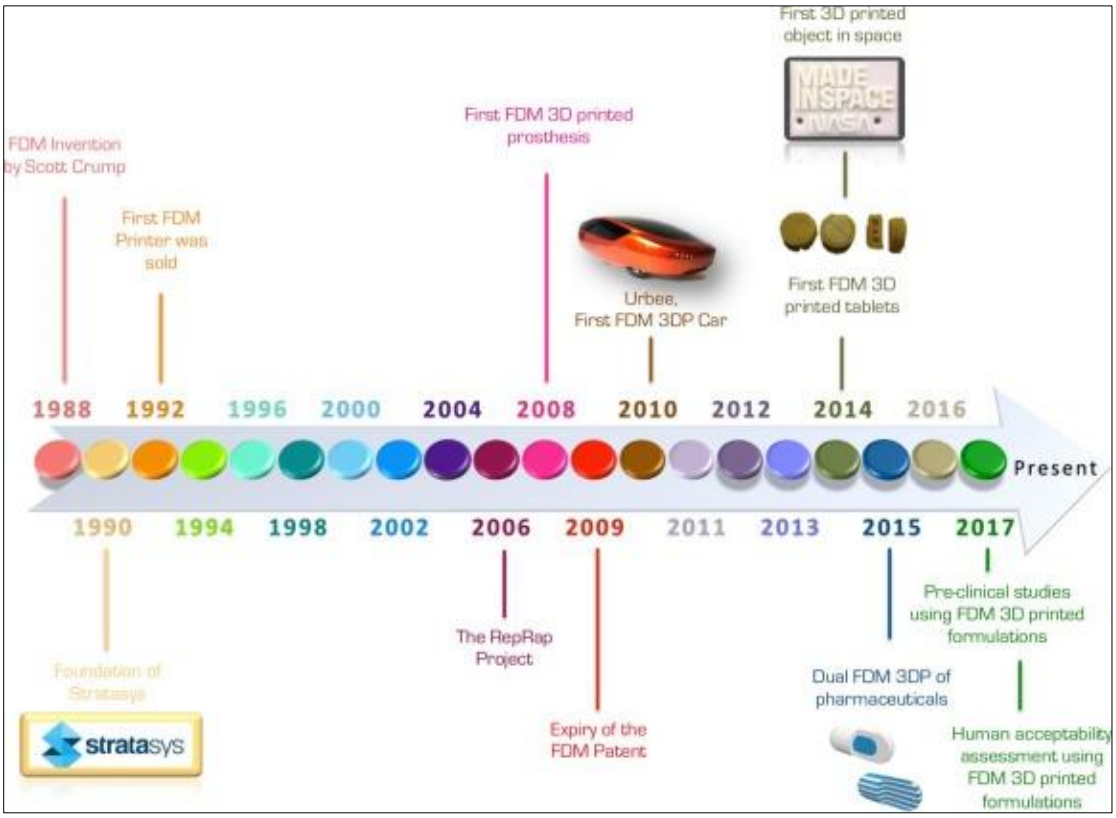

Figure 2 Timeline of the advances in 3D printed medicines [43]

4. Conclusion

3D printing is an amazing combination of chemistry, materials science and computer-aided design and manufacturing to create a brave new world. It is a versatile tool for realizing microfluidic chip holders. The flexibility of 3D printing enables rapid redesign of chip holders and customization for other chip geometries. The combination of silicon/glass microfluidic chips made with highly reliable cleanroom technology and 3D printed chip holders to connect the chip to the whole world is a promising solution for applications where biocompatibility, optical transparency and precise sample handling are ensured. 3D printing is a technology that has evolved from a niche market to widespread applications in the consumer market as well as in research and development institutions. Polymer science has contributed a lot as polymers are the material of choice when it comes to additive manufacturing.

The chemical industry is expected to play a very significant role in the explosive growth of the 3D printing business. Two global chemical giants – BASF and DuPont – have launched new materials to power 3D printers, forming strategic alliances in the fast-growing 3D printing industry. DuPont has introduced several high-performance materials in the form of fibres that 3D printers can use as raw materials. These are polyamides reinforced with glass or carbon fibres and combine lightness with excellent thermal and chemical resistance.

BASF has launched special photopolymers suitable for 3D printing. The company claims these polymers offer better mechanical properties and longer stability than materials available today. BASF went one step further and also partnered with 3D printer manufacturers, seeking synergy between materials expertise and 3D printing software. The availability of such new materials is an important step to meet the demands of component manufacturers who are looking for 3D printing materials with similar mechanical and chemical properties to known injection moulding polymers.

Additive manufacturing technology is used to create freely customizable items that are either in laboratory applications, such as custom and affordable reaction software, laboratory instruments that may have embedded sensors, or that are used in a chemical engineering context, such as upscaling methods. Applications include stirrer, packing structures and free catalyst design. Reactors are incredibly important in chemical engineering. The free design of the reactor enables 3D printing of functional prototyping, which expands the possibilities of reaction protocols and reactor optimization. Electrochemistry will certainly benefit from the production of such new devices through rapid prototyping, with

improved performance and at reduced cost. With the ease of testing new concepts and building prototypes, 3D printing is now increasingly exploring new opportunities for electrochemical applications in sensing, energy applications, and electrochemically assisted synthesis.

Compliance with ethical standards

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Disclosure of conflict of interest

No conflict of interest.

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