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Opportunities for process intensification technologies in nuclear effluent treatment: A review of precipitators, adsorbers and separators

G. Yaghy [a,b], A.S. Tonge [a,c,1], H. Abouhakim [a,d,1], R. Peeling [e], M. Talford [e,f], L. O'Brien [g], A. Paksy [g,2], P. Nevitt [g], F.L. Muller [a], B.C. Hanson [a], T.N. Hunter [a,*]

[a] School of Chemical and Process Engineering, University of Leeds, Leeds LS2 9JT, United Kingdom
[b] National Nuclear Laboratory, Culham Science Centre, Abingdon OX14 3DB, United Kingdom
[c] United Utilities Group PLC, Warrington WA5 3LP, United Kingdom
[d] School of Art, Design and Performance, Buckinghamshire New University, Buckinghamshire HP11 2JZ, United Kingdom
[e] Britest Ltd., Warrington WA4 4FS, United Kingdom
[f] Innovate UK, Swindon SN2 1FL, United Kingdom
[g] National Nuclear Laboratory, Chadwick House, Warrington WA3 6AE, United Kingdom

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ABSTRACT

This paper reviews the technologies and opportunities for process intensification (PI) in nuclear effluent treatment. PI is an area that has already created many innovations within the chemicals industry, and offers a growing field of research and development potential for nuclear operations. Technologies are considered here to be those ultimately resulting in step-change improvements to a number of operational aspects; such as smaller unit footprints, enhanced heat and mass transfer, reduction in secondary wastes, improved process safety and synergy, or direct integration with other downstream processes. Herein, we conduct a rigorous evaluation and scoping assessment of unit designs for the treatment of nuclear liquid effluents and solid-liquid waste management. Specific focus is given to precipitation, adsorption and separation operations, where over 250 articles are detailed, and prospects for technology transfer are discussed. In general, there is a trade-off in PI designs between operational simplicity (which may be more easily adopted in nuclear treatments) and more mechanically complex strategies that may, however, attain suitable scale-up requirements. Analysed options vary from those that would be radically different for industry, to those where applications are increasingly common in other process areas, with the advantages and limitations of all being discussed.

1. Introduction

The energy sector is facing one of its most eminent transitions in a century. The fossil fuel industry is now in a slow retraction phase, leaving its place to low-carbon energy. Amongst a number of alternatives, there is an international acknowledgement of the significant role nuclear power may play in climate change mitigation as a low-carbon baseload power source. For example, it is included in all four illustrative scenarios described by the Intergovernmental Panel on Climate Change (IPCC), which highlighted benefits of an increase in nuclear power capacity of between 60% and 500% by 2050. Indeed, as well as power, the potential role of nuclear in non-electric applications such as hydrogen production for transportation and heating is also recognised [1,2].

However, despite clear advantages, the high cost of new nuclear, as well as significant questions over its long-term waste management, have impacted on growth of the industry in the 21st century to date. To overcome these challenges, innovation in nuclear research and development is occurring across the whole fuel cycle, from designing new small modular reactors (SMRs) and advanced reactors for industrial applications, to a new generation of fuels and advances in recycling technology. Waste management of nuclear waste is of particular importance, as it enables the safe and responsible handling of radioactive materials, increases social acceptance of nuclear energy, and meets

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* Corresponding author.
E-mail address: T.n.hunter@leeds.ac.uk (T.N. Hunter).

[1] A.S. Tonge and H. Abouhakim contributed equally to this work.

[2] Current address: Jacobs Engineering Group, 305 Bridgewater Place, Warrington WA3 6XG, UK.

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the requirements of the international non-proliferation treaty. The UK's Advanced Fuel Cycle Programme (AFCP), a collaboration between the department of Business Energy and Industrial Strategy (BIES) and the National Nuclear Laboratory (UK), dedicates a great part of its investment to an integrated approach to used fuel and waste management [3–6]. One of the key aspects considered is the intensification of nuclear waste management to reduce the size of equipment or plants through modularisation and system integration, focussed on improved efficiency and economics. Miniaturisation of such operations will maximise the capacity of commissioned nuclear sites and associated lands, optimise the waste management process and lead to more controlled and efficient decontamination and eventual decommissioning [4,7,8].

While, currently, there is no single definition of process intensification (PI), it is generally taken as technological innovations leading to a step-change in overall process efficiency on chemical plants, such as significant reduction in footprint areas, often achieved with dramatically different design thinking. Over the last 20 years, it has been a frequent area of study for academic researchers and industry alike, driving better economic outcomes and reduced environmental impacts, while providing a holistic approach to cost-benefit optimisation [9]. On a similar basis, new equipment designs that provide PI, can be considered those that deliver critical enhancements in mass or heat transfer rates, while approaching a state where every molecule encounters the same processing experience. Such conditions result in greater control of reaction kinetics, higher energy efficiency and response times, as well as improved intrinsic safety within the process [10]. Maximising synergy between different processes is also of great importance, as it highlights the innate multifunctionality of PI. In addition, improvements achieved through PI can allow for either a combination of equipment into a single unit or the ability to take a more complete view to process integration, intersecting with optimisation techniques, such as multi-objective optimisation [11–14]. Modular systems have also been an area of interest in PI, as smaller equipment sizes lend themselves to be readily portable devices, giving benefits of flexibility and easier installation in space constrained sites. Nevertheless, there are issues around standards and costs to overcome (versus traditional economy of scale) in addition to mechanical complexity and lack of scale-up knowledge, although developments are addressing these limitations. Indeed, in many ways, SMRs can be considered *intensified* nuclear reactors, with the objectives to be innately efficient and economically viable through scale-out manufacturing methods, rather than scale-up.

More traditionally, PI research and development has been associated with industries such as fine chemicals and pharmaceuticals. To understand the potential utilisation of process intensified solutions for nuclear effluent treatment, it is pertinent to examine current treatment methods. Key techniques include precipitation, ion exchange, evaporation, reverse osmosis, ultrafiltration and solvent extraction [15]. For aqueous liquid discharges (beyond the direct fuel reprocessing cycle) the great majority of operations rely on co-precipitation and ion exchange, although some solvent treatments are used to remove caesium and strontium [15–17]. With co-precipitation, radionuclides are removed in the form of insoluble salts, such as metal hydroxides [18]. For ion exchange, they are exchanged with an anion/cation of an adsorbing material and then collected for disposal or decontamination [19,20]. Ultrafiltration membranes are also commonly employed in solids-liquid separation stages, along with more traditional gravitational processes [21,22].

Considering co-precipitation operations largely rely on continuous stirred tank reactors (CSTRs) there may be numerous benefits of taking an intensified approach to design. Typical inorganic crystallisations, while rapid, are complex reactions where the molecular variation in reaction conditions within CSTRs can lead to heterogeneous precipitated solids that are difficult to treat downstream. Being able to enhance mass transfer efficiency and ensure stable reaction environments will lead to more consistent waste products, with less need for onsite process control and potentially greater efficiency of radioisotope encapsulation (thus producing lower waste volumes). Regarding ion exchange columns, while operationally simple and flexible they are not materially efficient, and there is significant trade-off between working ion exchange capacity and process flowrates. This balance normally means that multiple columns are run in parallel, increasing plant footprint. Reducing plant size is also a critical concern for downstream solid-liquid separations, as neither membrane filtration nor gravity separation are effective at limiting required surface areas. Also, there is a growing interest in waste process integration, where effluent treatment and dewatering stages may be combined.

Outside of the nuclear industry, the application of PI technologies and evaluation methodologies to other industrial effluent treatment is an area that is now receiving increasing interest, with a strong potential for technology transfer. This is highlighted by the growing number of published papers on the topic. For example, there are >200 articles from the last decade published under combined topics of *Process Intensification + Effluents*, as categorised by Web of Science™, from a broad range of chemical and environmental science, agriculture and water management (see Fig. 1). As a further example, a review of PI for solid handling by Wang et al. [23], considered technologies for solids processing many of which would be relevant to effluent treatment, including precipitation, separation, granulation and milling. Similarly, a recent review by Coward et al. [24] discussed opportunities for PI in the UK water sector, containing a considerable overlap to industrial effluents, and that of Adamu et al. [25], on PI for carbon capture, presented applications for off-gas treatment that are similar in design to liquid adsorption columns.

While these previous reviews highlight a number of key, relevant technologies, there has been no previous comprehensive report into the application of PI for nuclear effluent treatment and waste management. Additionally, despite the large opportunity for PI within the industry, studies directly relating PI designs to nuclear waste operations are in some ways limited, although a great many of the articles summarised within this review can be directly applied. Amongst the notable examples that indicate the promise of PI in nuclear systems is the work of Flouret et al. [26], into the intensification of a stirred tank reactor-classifier for the continuous removal of strontium with co-precipitation of barium sulphate. More comprehensively, Bascone et al. [27], developed a novel flowsheet for the co-extraction of U and Pu in spent nuclear fuel processing, using intensified small-scale extractors coupled with model-based optimisation. Within the flowsheet, they included all geometrical variables, flow rates and reagent concentrations. Unit operations were reduced as were capital and operational costs, leading to considerable improvements in safety and equipment

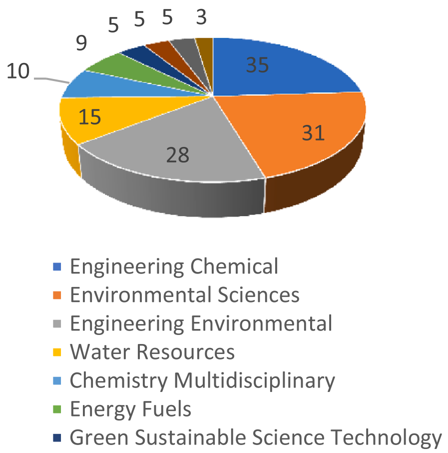

Fig. 1. Pie chart (as percent) detailing disciplines associated with +200 articles categorised by Web of Science™ over the last decade, that contain combined keywords of *effluents + process intensification.*

footprint. While this is related to fuel reprocessing specifically, rather than downstream effluent treatments, it highlights both the potential benefits of, and interest in, PI within the nuclear industry. In fact, there have been a number of important PI developments in nuclear fuel cycling that may also have uses within effluent treatment, such as the progress of centrifugal contactors as a central PI technology for liquid-liquid extraction, as recently summarised by Baker et al. [28,29].

Given the clear interest and relevance of PI to the nuclear industry, and the prospect for significant cost and space savings, this report provides a comprehensive review of related PI technologies that may be applied to nuclear waste management, in an attempt to address low current technology uptake in the area. In particular, we focus on prospective solutions for future advanced fuel cycle effluent treatments, comprising primarily of co-precipitation and/or ion exchange, along with associated solids-liquid separation. It is importantly emphasised that while there have been very considerable advances in the materials used for treatment operations in recent decades (such as advanced ion exchange resins) there has been much less development of the basic processes themselves. Emphasis in the nuclear industry has been, quite correctly, on safety and mechanical simplicity over innate efficiency. Yet, with the goal of the nuclear industry to be more sustainable, and with a large number of new treatment technologies being developed in other industrial areas, is this position appropriate for the future? Alternatively, is the natural conservatism in nuclear operations potentially holding back technology transfer? To help answer these questions, this review attempts to objectively consider what relevant intensified process options are available now and into the next decade, while comparing the relative advantages and limitations of each.

Technologies reviewed here are separated into three broad categories: Intensified precipitation reactors and mixers, intensified adsorption-contact units, and waste separators and classifiers. Some units are categorised as a combination of these types. A list of all the reviewed technologies is given in Fig. 2, which also highlights the designs that are considered to be a combination of classifications. Detailed descriptions of the technologies are given within the following sections, including an analysis of their possible application in nuclear operations. Additionally, Table S1 (presented within the Electronic Supplementary Materials, ESM) gives a summary of the techniques, along with assessed advantages, market readiness and ability to scale-up for effluent treatment applications.

2. Precipitators/reactors

Precipitation is the formation of solid particles from a solution. The extent of precipitation depends on the solubility of the solute, which in turn can be affected by various parameters such as temperature, pH, surface charge and mixing intensity. Precipitation operations, or more specifically co-precipitation, have been a primary method of nuclear effluent treatment since the start of the nuclear age. As evidenced currently at Fukushima, the flexibility of precipitation operations means they are still very much a critical decontamination technique and will likely remain so in future fuel cycles [30]. Precipitation can be intensified using various PI technologies such as microreactors, oscillatory baffled reactors (OBR) and spinning disc reactors (SDR). The common feature between these technologies is the intensification of the reactor's plug flow. Plug flow implies consistent shear or mixing rates through the reactor, and increased mass transfer rates compared to batch reactors, which promote the fast kinetic formation of crystals with low polydispersity levels (better homogeneity of crystal sizes) [23]. However, these reactors differ with respect to their functionality, mixing dynamics and mechanism by which intensification is achieved, causing a large variation in operating conditions, residence times, their capacity/size and related scale-up opportunities. This means that not all are highly suitable for the nuclear industry, where maintenance and other internal-access dependant tasks may be unsafe, costly, or entirely unfeasible due to the hazardous nature of radioactive effluents.

For instance, one way to reach an intensified plug flow is to reduce the size of the reactor to a microchannel size, where the increased contact area to volume ratio leads to high mass transfer rates and micromixing from the innately small diffusion times. Microchannels are therefore very process efficient and the possibility of scaling-out has been considered for larger applications, although significant challenges remain in this respect. In contrast, using a conventional macroscale pipe reactor to achieve similar plug flow mixing conditions requires high flow rates, leading to unfeasibly long reactor lengths. Therefore, most *intensified* plug flow reactors consider static or dynamic methods to reduce reactor lengths, while keeping the benefits of consistent mixing and high mass or heat transfer. Intensification may be achieved by side flows, such as in the case of force-free helical reactors, or generating eddies through centrifugal forces, as in Taylor-Couette devices. Other type of reactors, in particular spinning disk rectors (SDR), also use centrifugal forces, but with short residence times that may require multiple reactors to work in series, or recirculation of the effluent. Oscillatory baffled reactors (OBR) and agitated tubular reactors (ATR), on the other hand,

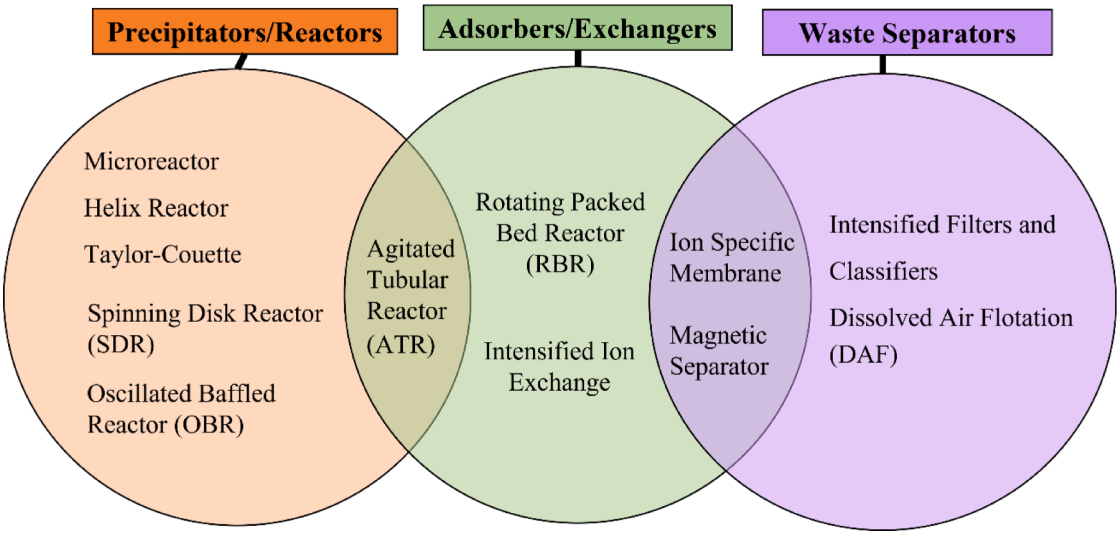

Fig. 2. Venn diagram showing the relationship between intensified reactors, adsorption contactors and solid-liquid waste separators considered within the literature review.

offer high radial mixing rates by mechanical oscillation while achieving residence times that are decoupled from reactor flowrates. Given this overview, the specifics of each reactor technology and its suitability for nuclear effluent treatment is discussed in detail within the following sections.

2.1. Microreactors

Microreactors or micro-capillary reactors strictly refer to reactors with a capillary channel or a network of channels with width and depth in the orders of tens to hundreds of microns and with a capacity in the microlitre scale [23,25,31] (see Fig. 3 for an example). As an extension, channel reactors with dimensions into the millimetre range are often considered within the same family of designs.

Microcapillary reactors offer highly controllable plug flow to carryout complex reactions, where the combination of multiple inputs can also be easily designed, which has generated a great deal of research interest over the last 15–20 years [33–45]. They exhibit high surface-to-volume ratios in a compact design [46,47], with significant reductions in footprint, and control over the heat and mass transfer, yield, selectivity, energy consumption and other derived product or process parameters. As an example, a flat microchannel with a width of 100 µm has a surface-area-to-volume ratio that is many hundreds of times larger than that of a 100 ml flask [43,47-51].

Microreactors have many features that may be advantageous for the nuclear industry as co-precipitation reactors. Due to their small size, operating conditions such as flow rate, temperature and pressure can be fine-tuned minimising safety concerns, which is unlike batch reactors where some parameters can only be adjusted after the end of each run consuming time and reducing the level of process control [25]. Additionally, over the last decade, microchannels and meso-scale channel reactors have been investigated for applications relevant to spent nuclear fuel reprocessing, radiochemistry and radionuclide separations, in particular, for liquid-liquid extraction [25,52,53]. Both single-component extractions of lanthanides, actinides or other metallic elements and multi-component mixtures have been recently examined by Tsaoulidis et al. [54,55]. The studies concluded that high interfacial area to volume ratio, and a large plug formation time, favoured by the small channel enhance the mass transfer and the extraction performance. A key finding was the high extraction percentage achieved in short residence times, where the small volume is compensated for by the high extraction efficiencies reducing waste accumulation, while the short residence times reduce solvent degradation.

A microreactor can be fabricated as a chip that offers integration of many processes (mixing, extraction, and phase separation) networked into a single device [54,56,57]. Such modularisation may be particularly advantageous for nuclear applications, considerably reducing overall radiation containment zones on plants and waste volume on decommissioning. Microreactors can also be fabricated with a broad range of materials such as glass, ceramics, perfluoroalkoxy (PFA), silicon, polymers and steel offering resistance to various factors including operating conditions (pressure, temperature and radiation) and physical properties of the reaction mixture, both of importance to nuclear processes where extreme conditions are expected [37,38,43,58-60].

A common concern about microreactors is solids handling, where particle size is limited by the channel width and may cause channel clogging or negatively impact downstream filtration processes [50, 61-64]. Several strategies have been developed to overcome fouling in microreactors. For example, Delacour et al. [65] introduced pulsed ultrasound to prevent blockage of micro-channels by solid particles. Alternatively, researchers have relied on relatively high velocities or on using straight channels (without any bends) to ensure that solids do not choke liquid flows. A more attractive alternative involves confining reactions to droplets that travel through the microreactor channels inside a carrier phase, and so away from the tubing walls [50,66]. For instance, Sen et al. [66], presented a microreactor-based method for direct uranium precipitation from loaded organics in continuous operation. Uranium removal > 99% was achieved for a residence time of 3 mins with an oil/aqueous phase ratio of 4:1. There was no need for a multi-stage operation leading to significant process intensification, as stripping of uranium from loaded organic typically requires a large number of stages.

Increasing microreactor capacity has been considered through either scale-up (using larger channels up to sizes that still preserve most the benefits of operating in small scales [67]) or more commonly scale-out (use of many equal size channels operating in parallel at similar conditions [54,68]). Nonetheless, the reality is that for a nuclear effluent treatment process or indeed a fuel reprocessing facility, where capacity may be in the range of hundreds of m^3/day, thousands of individual microchannel reactors would be required [69]. Presently, there is no clear example of industrial application at this scale, where even the level of flow control between reactors would be extremely complex, and in many ways mitigate the natural simplicity of the reactor designs. Therefore, it may be that other more scalable designs, as detailed below, may offer greater potential for direct industrial utilisation in the near future.

2.2. Helix and Taylor-Couette reactors

On a larger scale than the microchannel reactor, helical mesoscale reactors, also called Helix reactors, offer an intensified alternative to a straight pipes [70]. Using a coiled tube arrangement, additional Dean vortices are created to enhance radial turbulence [71]. The flow profile in a helical coil is not parabolic and due to centrifugal forces, a secondary flow is generated in which two counter-rotating Dean vortices are formed perpendicular to the direction of flow. These vortices cause high levels of radial mixing that results in a narrow residence time distribution inside the reactor and greater shear, as well as a larger pressure drop [72]. Compared to straight tubes or batch reactor of similar capacity, Helix reactors provide enhanced heat and mass transport due to their controllable flow patterns and high surface to volume ratio [72–75]. Different geometries can also be used to modify flow and

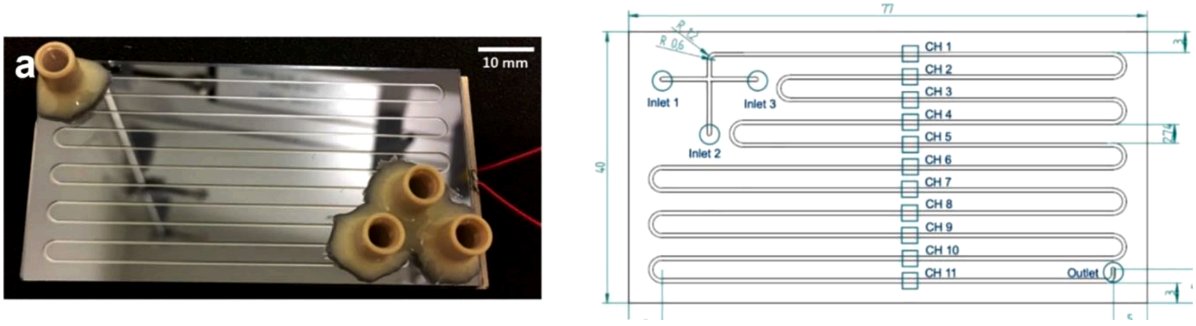

Fig. 3. a) Photograph of a microreactor for particles synthesis, and b) Sketch of the microchannel structure (reproduced from [32]).

mixing patterns, from singular straight helical coils to multiple angled coils, known as coiled flow inverters, as presented in Fig. 4 [74].

In terms of relevant inorganic precipitation studies, continuous precipitation of calcium carbonate has been investigated in a modular coiled flow inverter (CFI) made of polyvinyl chloride (PVC) tubes ($d_i =$ 3.2 mm) with slug flow patterns [74]. Conversion reached 90% and a narrower particle size distribution with uniform morphology was obtained as compared to a batch reactor. Also, inline video-imaging has been used by Wiedmeyer et al. [76] for crystal shape analysis and residence time estimation of potash alum using a similar helically coiled flow tube (HCT) crystalliser. Their main findings were the size-dependant particle residence time, where larger particles move faster through the helical reactor than smaller particles. Consequently, small crystals have more time to grow leading to higher uniformity in output particle size which can be desirable for downstream separation.

Despite these studies, helical reactors have not yet been studied for spent fuel recycling or effluent treatment, while they present an interesting opportunity for the nuclear industry. It is a simple and reliable reactor with passive mixing and absence of moving parts or baffles which reduce failure rate and maintenance that is particularly undesirable in the nuclear industry. In addition, synthesised or precipitated particles experience a narrow residence time distribution and mixing conditions that creates uniform supersaturation, thus size distributions are generally very small, leading to better control [77]. Helical reactors also provide the opportunity for tunable residence times due to its plug flow characteristics, where a balance between conversion rate and residence time should be assessed in a radioactive environment to prevent solvent and adsorbent degradation [53]. It is also noted that these mesoscale designs, while being more scalable than microchannel reactors, still suffer from relatively poor scale-up capability that would limit their use in large volume plants. Quoted flowrates for related photocatalyst reactions with single helical tubes are < 0.25 l/min [62], so for effluent treatment, many tube reactors would still be required in parallel.

An alternative mesoscale and baffleless secondary-flow reactor design is the Taylor-Couette reactor (TCR), sometimes referred to as a vortex flow reactor. It consists of a cylindrical shell in which an inner cylinder is inserted so that a customised annular gap is formed and the cylinder and/or shell can counter- or co-rotate at different rotational speeds. Various flow regimes can be set in the annular gap by varying the rotational speed/directions of the cylinders and the annular gap. The flow can be then tailored specifically to the demand of the process from Taylor-vortex flows, formed from the interaction with the rotational cylinder up to complete turbulence, as seen in Fig. 5 [78–83]. While this geometry is advantageous for nuclear applications, the moving parts of the Taylor-Couette make it a less appealing option than technologies with passive mixing like the helical reactor.

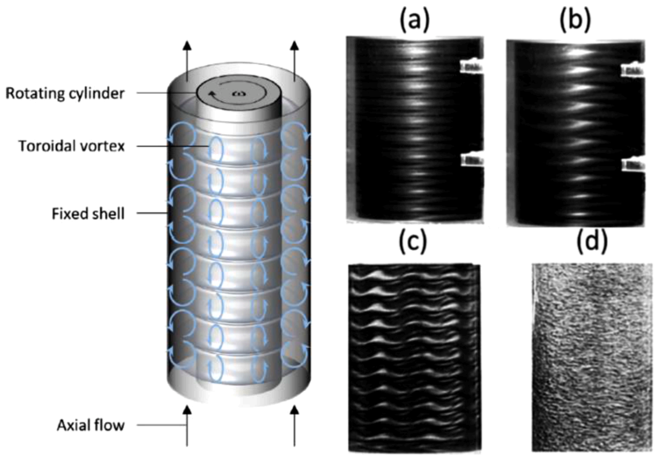

Fig. 5. Sketch of Taylor-Couette Taylor with a fixed shell and a rotating inner cylinder. Various flow regimes are shown as follows: (a) Taylor-vortex flow, (b) Wavy vortex flow, (c) modulated wavy vortex flow, (d) turbulent flow (reproduced from [82]).

One key advantage of this form of mixing is that it opens up the possibility of achieving a long residence time with plug flow in a compact batch-like geometry. Each vortex created is essentially a stirred tank in the sense that it is well-mixed throughout its volume. If operated correctly, such reactors can therefore operate as a series of well-mixed volumes, resulting in plug flow in a similar manner to CSTRs in series [10,82,84]. High heat and mass transfer coefficients are achievable in TCRs, due to the good radial transport engendered by the vortices and high surface to volume ratio [10]. TCRs also offer high uniformity of shear forces within the annular gap, which leads to well dispersed suspensions and offers great advantages for crystallisation/co-precipitation purposes, allowing fine tuning of crystal/particle size distribution [85]. The relatively open flow structure of TCRs additionally lends itself to applications involving solids up to high loadings, as it is not particularly prone to blockages.

Industrial applications of TCRs are still limited by the complexity of the flow regimes and, again, difficulty of scale-up, especially when considering the seals, bearings and mechanical drive required to move a large-scale rotor. Academic applications on the other hand are various, and include amongst others, solid processing operations. For example, TCRs have used in several crystallisation studies [86–89] where required residence times have been reported to be 3–5 times shorter than in conventional CSTR crystallisers. The size distribution of crystal product was also much narrower in the TCR when compared to the conventional crystalliser (size distribution coefficient of 0.45 and 0.8 respectively) [89]. Fluid dynamics studies have also been performed in order develop

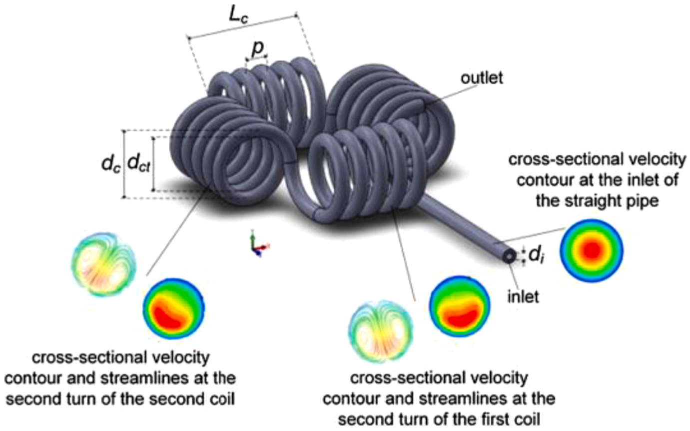

Fig. 4. Design and representative flow contours from a multiple angled coiled flow inverter frame (reproduced from [74]).

a phenomenological model as a tool for the design and production of particles with controlled properties [90].

TCRs have also been studied for effluent flocculation reactions. Flocculation of bentonite clay was performed in a turbulent TCR under various shear rates by analysing aggregate morphology parameters like the radius of gyration and circularity [91]. The unique ability to radially inject fluid into the rotating annulus, was also used to investigate how specific hydrodynamic flow fields affect assembly and structure of aggregates during the flocculation process [92]. Faster floc growth rates and decreased floc fractal dimensions were observed for higher order flow states, indicating improved mass transfer of the polymer flocculant and breakage at the edges of the flocs, respectively. These studies highlight the TCRs relevance for nuclear effluent treatment and solids separation, although, the mechanical complexity of running a number of these units in parallel may limit their potential.

2.3. Spinning disk reactor (SDR)

The spinning disk reactor (SDR) intensifies mixing by high centrifugal acceleration of liquids flowing over its surface. Due to their distinct geometry, SDRs produce very different characteristics and processing conditions compared to other centripetal flow reactor designs, i.e., the Taylor-Couette reactor. In SDRs, the solution is fed into the top of the reactor at the centre of its disk surface, and the liquid then forms a highly sheared thin film (~50–500 μm) with high surface area to volume ratio (typically up to 30,000 m^2/m^3). Film thickness is dependant on a range of operational parameters, such as disc speed (between 200 and 60,000 rpm) and diameter, liquid flow rate (0.5–200 ml/s), disc temperature, reactor pressure and physical properties of the liquid (density and viscosity). A 500 mm disk can typically process ~150 kg hr^{-1} of liquid with similar viscosity to water [93–98]. Good temperature control of the disc surface is typically achieved by a heat-transfer fluid circulating underneath the reaction surface meaning that the reactor can also be used in drying applications. Schematics showing the operation of an SDR are given in Fig. 6 [99,100].

SDR enhance crystallisation/precipitation due to the very high heat and mass transfer associated with thin films. Similar to microchannel reactors, reduced path lengths in the thin films increase conduction and diffusion through the liquid with larger transfer coefficients observed at higher disc speed and liquid flow rate [97,101-104]. Low film thickness combined with relatively high radial velocity film give rise to very high shear stresses/shear rates within the liquid film. The high shear rates would be very advantageous for processing shear thinning fluids, such as co-precipitate sludge in the nuclear industry, where the shear may decrease the fluid viscosity and ease flow and materials handling. In such solid-liquid applications, SDR is also resistant to fouling, due to the open surface with the additional possibility of using Teflon discs (instead of stainless steel or brass) to decrease adhesion and enhance resistance to reactor materials degradation [105].

The SDR has the ability to achieve high supersaturation levels via rapid micro mixing in the thin film, a performance benefit that stirred tank reactors cannot provide at the same level [106,107]. In particle synthesis applications, SDRs have been shown to be able to produce fine crystals within a tight particle size distribution, while significantly increasing the reaction speed (99.9% reduced compared to a batch process [105]) with related reductions in impurities and reactant inventory. These characteristics make the SDR particularly attractive for various nuclear industry applications.

While SDRs haven't yet been used specifically for any nuclear waste treatment purposes, it has been studied extensively across other particle science fields in pharmaceuticals and fine chemicals production, for various purposes, such as polymerisation [94,108-110] organic crystallisation [95,105,111], food processing [112] and preparation of many kinds of nanoparticles [113–116]. A recent example of an SDR application that is transferrable to nuclear effluent treatment is the study performed by Vilardi et al. [97], who investigated the performance of an SDR for the removal of nitrate from aqueous solutions, using iron nanoparticle production as a reducing agent. In this study, the SDR was able to overcome the mass transfer limitations that characterise photocatalysis processes when adopted in large-scale equipment [117], where the use of zero valent ions is increasingly seen as an important technique in water treatment [118].

One of the unique characteristics of SDRs is the extremely short and controllable residence times, of the order of seconds, rather than the minutes or even hours required in conventional reactor configurations such as large CSTRs. While the short residence times are matched by the considerably faster mass transfer rates, for kinetically slow reactions, this can result in the requirement to use multiple reactors in series (or recycle in a number of passes) to gain the required overall reactor residence times. Related to this is the trade-off between faster rotation rates, which increase intensification and reaction efficiencies, and the corresponding reduction in residence time. The lack of independent residence time control is one significant factor that has held-up industrial implementation of this design, as well as the relatively high cost, limited availability and lack of awareness of the technology [93]. Nevertheless, as noted, industrial scale-up of SDRs is much easier than with microchannels or mesoscale reactors, with accessible flowrates up to 12 l/min for 1 m discs [119], although there are restrictions on how

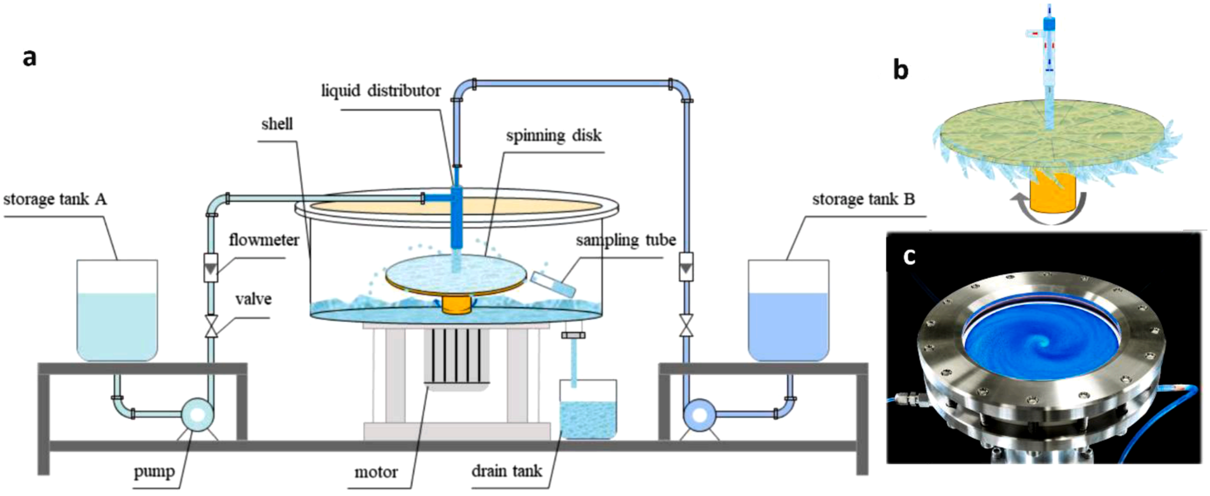

Fig. 6. a) Schematic of a spinning disk reactor experimental setup and b) a close up of the spinning disk (both reproduced and modified from [99]). c) An image representation of a SDR (reproduced from [100]).

much process throughput rates can be varied.

2.4. Oscillatory baffled reactor (OBR)

The oscillatory baffled reactor (OBR), as illustrated in Fig. 7, is perhaps the most well-known type of oscillatory flow reactor [120,121] designed to fully de-couple the method of intensified mixing in plug flow from the bulk flow rate. It consists of a tube reactor fitted with periodically spaced baffles, having an oscillatory motion (range 0.5 to 10 Hz) creating vortices superimposed upon the net flow of the process fluid [23,24,122,123].

The constant creation and destruction of vortices in OBRs results in a series of well-mixed stirred reactors, on either side of the baffles. As the degree of mixing is independent of the net flow, plug flow is then achieved with a minimum Reynolds number (laminar flow). Due to this unique flow characteristic, OBRs can offer then what conventional tubular reactors cannot, a greatly reduced length-to-diameter ratio reactor (100-fold reduction in reactor size) and longer residence times, which provides a reduction in footprint [10,125,126]. The vertical mixing patterns also enhance heat and mass transfer by increasing surface area to volume ratio (Nusselt number, Nu) is typically enhanced by a factor of 10–30, with mass transfer is reported to increase by ~75% [127–130]. In terms of safety aspects, critical to the nuclear industry, there could be improvements due to the smaller reactor size (and thus controlled access area) and lower inventory of materials[125,131].

For particle or polymer synthesis, OBRs result in better distribution of reagent supersaturation, enhanced temperature and cooling rate control, as well as consistent product properties such as floc size, morphology and size distribution. These properties have led to improved particle flow characteristics and have a direct impact on downstream processes such as filtration [132]. The uniformity of mixing in OBR has also been shown to be advantageous when handling shear sensitive materials also such as crystals and flocculators [133,134], as the mixing mechanism is similar at laboratory and industrial scale (assuming a consistent gauge pipe is used) scalability of OBR is another distinct advantage as compared to previous reactor designs mentioned. OBR technology is also mature and has been applied in industries such as biodiesel productions [135] and bioprocessing [136]. At an industrial level, a demonstrator OBR was also installed at the Sanofi pharmaceutical crystallisation plant in Haverhill, UK, which led to a significantly smaller reactor size when compared to CSTRs of similar throughput (reactor volume was reduced by >99% against the traditional approach) [137].

Indeed, there are a number of previous examples of the use of OBRs for the synthesis or crystallisation of active pharmaceutical ingredients (APIs), such as Lawton et al. [132], who investigated continuous crystallization in an OBR, finding the processing times were on average 80% shorter than batch processing. Similarly, McGlone et al. [126] investigated the efficacy of continuous crystallization of APIs in OBRs, highlighting the efficiency gains in moving from traditional slow batch mode crystallisers. Additionally, Abernethy et al. [138] carried out L-glutamic acid crystallisation in a meso-OBR and achieved a narrow crystal size distribution with particle sizes of ~ 20 μm. In terms of examples of more significance to effluent treatment applications, Ni et al. [133] investigated the flocculation of bentonite in an OBR and concluded that the oscillation amplitude is the key parameter affecting the percentage of flocculation. More recently, Castro et al. [139] considered the inorganic co-precipitation of hydroxyapatite in a scaled-up meso-OBR, where due to the fast kinetics, the best performance (in terms of size distribution and consistent morphology) was found for smaller residence times. OBRs have also been used as reactors to produce materials suitable for ion exchange in nuclear applications. Grimaldi et al. [140] investigated the use of an OBR for an industrial scale intensified production of zeolite-A, while Laybourne et al. [141] combined oscillatory flow reactors with microwave heating for the intensified production of metal organic frameworks.

One critical aspect of OBR performance, when considering co-precipitation applications, is the mixing dynamics of solid-liquid systems, and the related limitations of OBRs with respects to fouling. Due to the multiple baffle arrangements, optimisation of the pulse-flow rate for large particle sizes is difficult, while hold-up of solids in the reactor is a complex process to predict. Four distinct flow regimes have been observed in solid-liquid suspensions processed in OBRs, based on the relationship of the oscillatory axial velocity to the hindered settling velocity of particles, input power and the concentration ratio between adjacent cells [142]. These complex dynamics lead to differing degrees of retention of solids in mixing cells, and often is also dependant on the free baffle area (α) which in general, leads to longer residence times for particles than the bulk fluid [120] well as fouling deposition, if mixing is not carefully controlled. However, a high degree of mixing through oscillation can enhance the flowability of solid particles and OBRs have proven capable of handling solid suspensions at high solids loading (~10%) especially in organic crystallisation applications [143,144]. Nevertheless, the higher density of heavy metal co-precipitate crystals used in effluent treatment may accentuate the propensity for fouling.

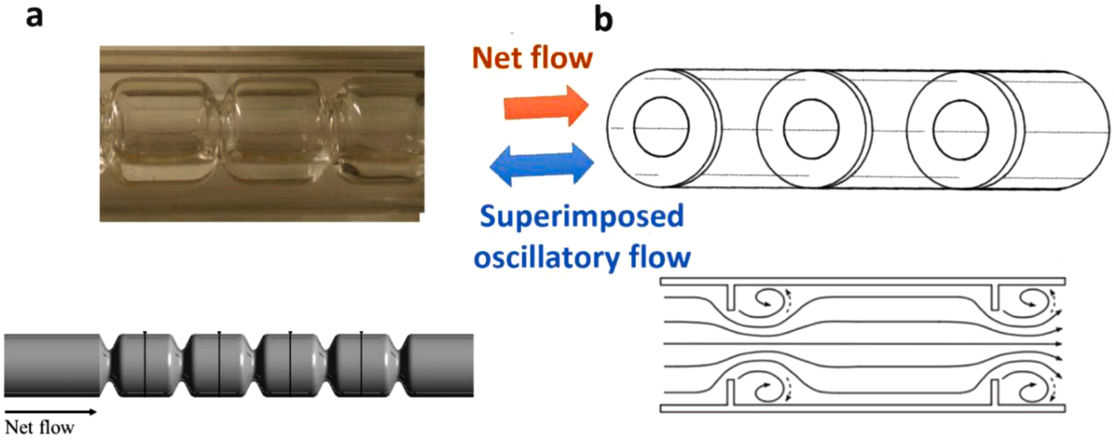

Fig. 7. a) Photograph of mesoscale OBR and its geometry simulated by CFD (reproduced and modified from [124]). b) Schematic of an orifice plate geometry OBR and sketch of eddy formation (reproduced and modified from [120]).

3. Combined precipitators and adsorbers

3.1. Agitated tubular reactor (ATR)

An alternative oscillating intensified plug-flow reactor, which has been designed for better incorporation of solids with reduced fouling, is the agitated tubular reactor (ATR), such as the AM Technology Coflore® reactor (shown schematically in Fig. 8a) which is primarily aimed to optimise multiphase mixing/contact operations in continuous solids catalysed chemical reactions [145]. Similar to an OBR, it decouples lateral shear from plug-flow throughput rates using oscillations, allowing residence times to be increased without compromising mixing. In contrast, it employs an open internal mechanical agitator bar, which generates shear from pulse oscillations (in a frequency range of 1–6 Hz) in the radial direction (rather than in the streamwise direction like an OBR). This configuration reduces internal recirculation from the baffles and the potential for particle deposition. It consists of a configurable set of modular tube sections that contain an internal, free-floating agitator bar driven by controllable external pneumatics. There has been increasing research interest in the use of ATR type devices, and smaller agitated cell reactors (ACRs), which have demonstrated effective scale-up [146–148]. Specifically, the pilot-scale Coflore® model has variable volume capacity enabling process flow rates of up to 30 l min^{-1} per unit (depending on number of tubes used as residence time) [145,149].

As the radial agitation is mechanical (depending on the momentum interaction between the outer tube and the agitator bar) the mixing characteristics are essentially independent of throughput, making the equipment well suited for multiphase mixing and processing [150]. The mixing dynamics of the ATR are complex; yet, they have been characterised both experimentally and numerically using computational fluid dynamics simulations [145,150,151]. It has been shown that the lateral mixing leads to greatly enhanced mass transfer rates, while maintaining adequate suspension lift to ensure good dispersion of 50–100 μm dense particles, possibly improving both co-precipitation kinetics and solids mixing. Similar to the OBR, most previous research using ATRs (and the smaller ACR) involving particles has been focused on solids catalysed organic synthesis or organic crystallisation [146,152,153]. Nevertheless, some notable recent investigations highlight application areas with inorganic co-precipitation. Chuzeville et al. [154], used an ACR as an intensified eco-friendly process for the synthesis of amorphous calcium carbonate nanoparticles, while an ATR was utilised by Tonge et al. [148] as a co-precipitation device for iron hydroxide coagulation. In this work, the focus was to remove common anionic dyes, through the combined use of nano-adsorbents along with coagulation enmeshment. Therefore, while the application area was not nuclear specific, the design and operation of the ATR is consistent with expectations for its use to remove heavy metals.

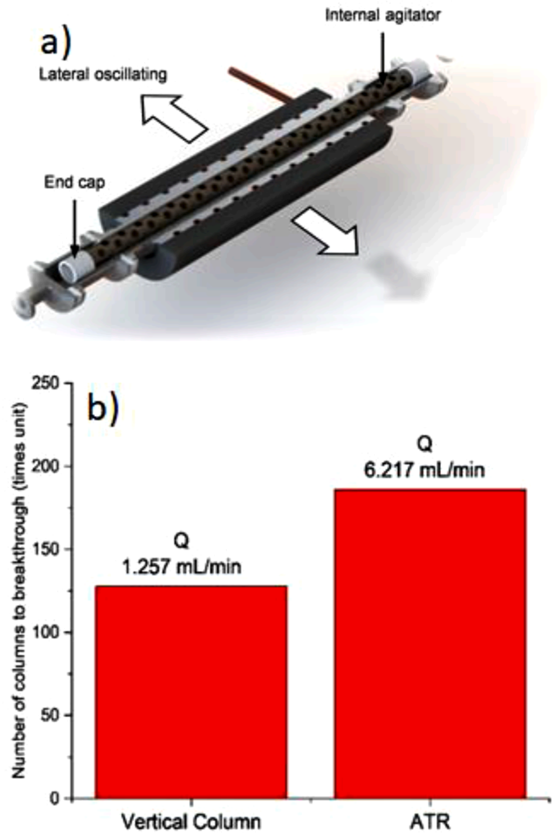

Fig. 8. a) Schematic of an agitated tubular reactor (ATR) showing lateral oscillation direction (reproduced from [150]). b) Comparison of ion exchange between a vertical elution column and an ATR for strontium removal (reproduced and modified from [155]).

Given this work, it is evident that designs like the ATR could be used as precipitators in effluent treatment. However, other current research also suggests it could act as an intensified ion exchange unit (hence presenting it a combined unit design). Specifically, Yusuf Prajitno et al. [155] considered the use of an ATR for large granular ion exchange resins (see Fig. 8b)). In the conducted work, the agitator bar within the ATR tube was filled with ion exchange resin (which was kept in place using sieve mesh around the agitator). In this way, instead of liquid eluting down through a vertical column (as in a regular ion exchange system), effluent was pumped through the ATR. The agitation aided mixing contact between the ion exchange resin and effluent, enhancing adsorption kinetic rates and increasing relative exchange performance by 30%. Such increases would result in substantial cost savings industrially, from reduced resin volumes. Nevertheless, one possible downside of the current ATR design is the difficulty in replacing the ion exchange agitator bar, in comparison to pumping exhausted resin out of columns, as is often performed for ion exchange in nuclear effluent treatment [156]. It may be that an alternative mechanism is required for the ATR to better enable safe, efficient resin replacement if utilised this way (e.g., using automated dispensers to load/unload charged ion exchange pellet tubes).

4. Adsorbers/exchangers

In addition to focusing on co-precipitation operations, which most of the reactor designs outlined in Sections 2 & 3 would be utilised for, this review has also considered methods to enhance adsorption processes, as alternatives to batch contact tanks or static ion exchange columns. Indeed, as evidenced by the use of the ATR discussed above, there are theoretically significant increases in process efficiency that may be achieved, with related reductions in unit footprints. While, in general, designs for intensified contactors are not as common as chemical reactors, it is an area of increasing industrial interest.

4.1. Rotating packed bed (RPB) contactors

One flexible technique that has been applied in a number of areas is rotating packed bed (RPB) contactors (shown schematically in Fig. 9). RPBs replace the gravitational feeds of conventional packed beds with a high-gravity environment (100–500 g) via the action of centrifugal acceleration to enhance mass transfer contact within the bed. Similar to the underlying principles of spinning disc reactors, the centrifugal field forms sheared thin films, giving a higher specific surface area compared to conventional packed ion exchange or bed contactors and can considerably improve mass transfer processes [157–162]. Compared to conventional packed beds, RPBs reduce footprints (up to 10 ×) [163], improve efficiency with lower absorbent concentrations and higher mass transfer coefficient (2.7 × higher) [164–166], as well as enhance

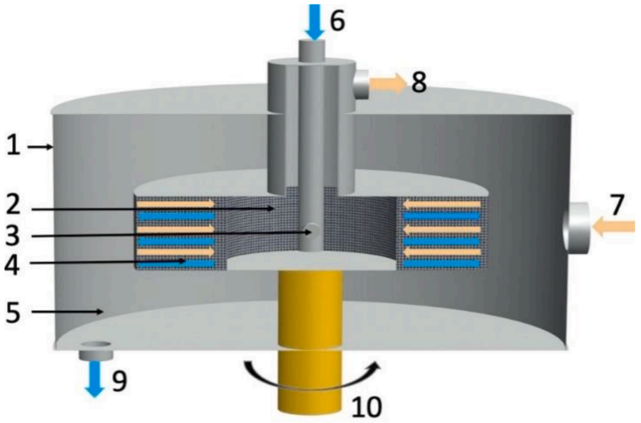

Fig. 9. Schematic of a typical RPB with a single block counter-current flow arrangement (1. Casing; 2. Inner cavity zone; 3. Liquid nozzle; 4. Packing; 5. Outer cavity zone; 6. Liquid inlet; 7. Gas inlet; 8. Gas outlet; 9. Liquid outlet; 10. Motor) (reproduced from [168]).

selectivity [163,166]. RPBs overall may provide significant improvements to separation processes and expand the operating window of treatable effluents, especially highly viscous fluids [25,167].

RPBs have been mainly employed with gas-liquid separation and distillation processes [166,169-173], and three-phase wastewater treatment operations, such as air-stripping [23,174]. They are also increasingly being utilised as two-phase solid-liquid bed contactors, for the removal of mobile contaminants, such as dyes, heavy metals and fertilisers [175–177]. For instance, Hagag et al. [178] applied a RPB for the removal of copper ions from industrial wastewater, where it was demonstrated that the exchange system was able to provide very high yields in batch operation. Although no direct comparison was made to traditional ion exchange systems, a correlation was provided to enable scale-up of the system with work ongoing to extend to continuous operation. A similar study performed by Wu et al. [179] for removal of ammonium from both distilled and pond water, indicated the feasibility of the technology and found clinoptilolite (also commonly applied in nuclear operations) enhanced removal efficiency, compared to a granular zeolite form.

The downside to using centrifugal force, is the small residence times for high rotational speeds (again, as per SDRs). Therefore, for normal ion exchange operations (with column residence times of 10–30 mins) multiple passes through the RPB contactor would be required if operated in a continuous fashion, even when considering the increase in mass transfer efficiency. Further, the mechanical stress associated with the high-speed rotation increases the level of equipment maintenance; although, it is perhaps noted that it is common in many cases of intensified designs that they are functionally more complicated than traditional units.

4.2. *Monoliths and non-mechanical intensification of ion exchange*

As well as using intensified mechanical contractors, such as ATRs and RPBs (as discussed) ion exchange processes may also be intensified through the use of monolith supports, taken from their use as chemical catalysts. Monoliths usually consist of structured honeycomb capillary channels, upon which the catalyst is coated within a wash coat layer of porous material and sintered. Monolithic catalysts were initially developed for use in automotive exhaust emission control but have gained a more diverse range of applications in the chemical and allied industries [93,180] as their manufacturing techniques (e.g., through 3D printing, see Fig. 10) have become more cost effective [181].

Monolithic contactors offer the advantage of overcoming mass transfer limitations without the need for additional mechanical energy to mix or spread the reacting fluids into thin layers with a high interfacial area. Therefore, they lend themselves to miniaturised process equipment with much lower power inputs [184]. Typically, the channel side length in monoliths is of the order of 1–5 mm and reactions occur at catalytic or exchange sites in pores of the order of tens of nanometres. These small length scales can be contrasted with the much larger sizes in traditional reactors on the scale of several centimetres/meters. By minimising the transport and diffusion distances, they bring the reactants together within intimate contact enhancing mixing within the channels [93,185,186].

While the use of catalyst monoliths can bring added functionality to small capillaries and channels, the same is also true for their potential use to intensify ion exchange, where they can be embedded in tubular reactor designs [182]. Monolithic contactors thus are a path towards modularisation and are innately flexible, enabling the replacement of large, centralised exchange columns with smaller reactors/contractors

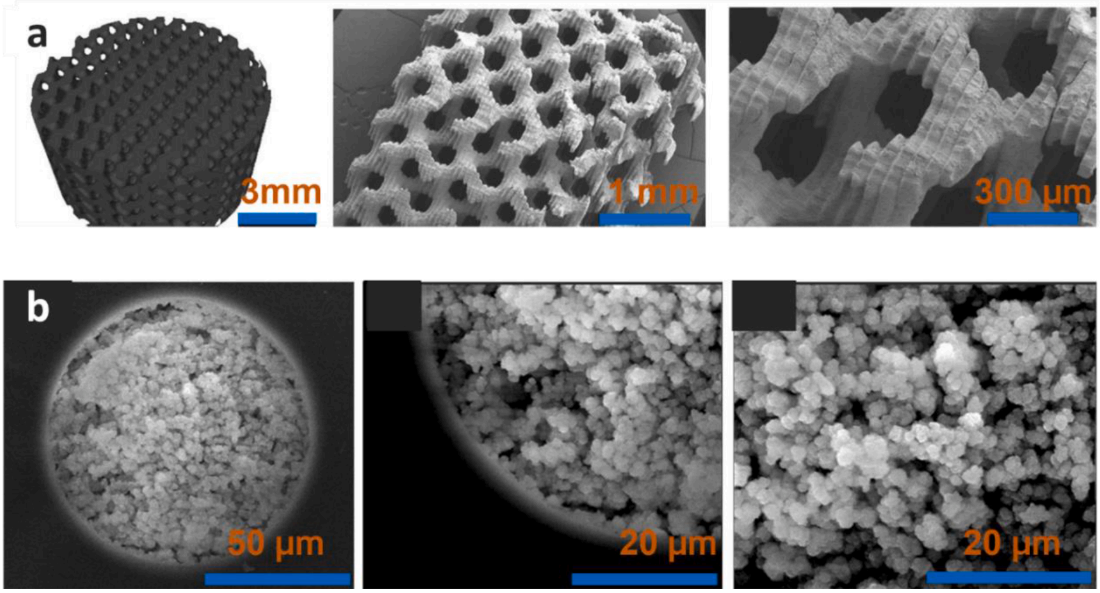

Fig. 10. a) CAD model and SEM images of a monolithic adsorber for anion exchange and wall thickness of 500 μm (reproduced and modified from [182]). b) Representative SEM images of a monolithic column prepared in a 100 μm diameter capillary (reproduced and modified from [183]).

[187]. These portable plants could be easily transported to the locations of different feedstocks, a highly desirable degree of mobility for the nuclear industry. Like many non-mechanically intensified plug flow reactors, monolith contactor scale-up (or rather scale-out) is relatively easy conceptually, based on the assumption that hydrodynamics and reaction in a single channel is the same for all channels within the reactor. The cost of the monoliths is a major consideration (despite modern advances) and the recycling or reusability of monoliths would be limited with radioactive contaminants.

Monolith catalytic reactors have been applied to various applications, such hydrogen production from steam reforming [188], manufacture of synthetic fuels using Fisher-Tropsch (FT) synthesis [93,189,190] and the purification of air or water [117,191]. However, they have also been used as intensified ion exchange materials for effluent decontamination. For example, the use of monolithic micro-porous polymers has been studied for the removal of nickel ions, improving efficiency compared to a regular packed column by 80%, by maintaining a high internal liquid phase volume, resulting in smaller pressure drop with a large surface area available for adsorption [192]. More recently, Simon, Dimartino and co-workers have developed 3D printed monoliths as both anion and cation exchange materials for use in well plates to remove proteins [182,193,194], while Halevi et al. [195] used 3D printed zeolite monoliths for Cs^+ and Sr^{2+} removal, in direct nuclear effluent treatment applications.

Additionally, there has been significant work in the use of monolithic porous polymer foams synthesised from high internal phase emulsions (polyHIPEs) that can be used for ion exchange columns for heavy metals removal when functionalised [196–198]. In terms of other recent examples, functionalised polyHIPEs have been used to remove metals such as boron [199] and lead [200], while their efficacy for heavy metals removal has also been improved through magnetic functionalisation [201]. Perhaps most notably, they have also been functionalised for Pu specific adsorption by researchers at the Savannah River National Laboratory and University of South Georgia. Here, the monoliths were found to eliminate the slow diffusion through resin beads commonly seen in traditional systems and also increase ion exchange site accessibility [202].

Ion exchange has additionally been intensified by combining with electrodialysis, in a hybrid process known as electropermutation [203,204]. Here, the application of electrolysis helps drive adsorbed ions from the exchange beads, continuously regenerating the material. While it was first proposed many decades ago, it has only more recently gained industrial interest. The process has been used to capture various heavy metals, such as nickel [203] or lead and cadmium [205,206], while caesium has also been separated and concentrated [207], of direct relevance to nuclear effluent applications. In related studies, Abdel-Aziz et al. [208], considered the use of ion exchange to enhance electrochemical separation of copper in a fluidised bed reactor, showing that the combined process led to considerable improvements in the decontamination factor along with a drop in relative energy consumption. In general, the use of non-mechanical ion exchange intensification offers a viable, technologically mature route for integration in nuclear effluent systems, where benefits may be further realised if combined with other intensified reactor designs. Also, they offer a convenient route to incorporate other methods to improve ion exchange efficiency, such as pre-activation with acids or salts, which has been shown to be particularly effective to improve zeolite performance for Cs^+ and Sr^{2+} removal [209,210].

5. Combined adsorbers and solid-liquid waste separators

One critical aspect of effluent treatment that is often overlooked, is the efficiency of downstream waste separation operations. This is particularly important for nuclear effluents, owing to the requirement to consolidate radioactive wastes effectively for eventual disposal, using methods such as thermal treatment or encapsulation. Potentially, the combination of precipitation or adsorption reactors with separation stages would offer process efficiency benefits, as well as significant footprint reduction. In general, by waste separation, we refer here to solid-liquid dewatering and consolidation (especially of precipitation sludges) and not the separation of U or Pu from fuel recycling operations. In terms of combined adsorption and separation systems, we also consider physical separation of ions using membranes, owing to the possibility of using them in combination with solid separation in ultrafiltration systems. Magnetic nanoparticles are also discussed as combined adsorbers and separators, owing to their unique promise to be used as high performance, high surface area adsorbers that can also physically separate solid waste with relative ease.

5.1. Ion selective composite membranes

Membrane systems that separate on the basis of size exclusion are routinely used within the nuclear industry and are not considered part of this review. Ultrafiltration systems, in particular, are commonly employed to separate particle rich streams in nuclear effluent treatment, where often, the majority of the radiological activity can thus also be removed, as it is concentrated in the solids phase [22,211,212]. Examples include the Enhanced Actinide Recovery Plant (EARP) at Sellafield in the UK, which use crossflow filter designs to separate out activity rich hydroxide precipitate sludge, allowing the liquid effluent to be discharged [69]. Membranes with smaller exclusion sizes, such as reverse osmosis units, are less commonly employed [22]. While they can be used to remove radioactive ions from the liquid phase, in addition to particles, they are a very energy intensive process. It is also noted that, based on size exclusion alone, they are non-selective, and thus, not an efficient way to remove small ppm levels of radioactive contaminants from effluents that may contain high overall levels of electrolytes.

Currently, as an alternative, there is a lot of research interest in intensifying membranes through the generation of chemical or physical selectivity for particular species, enabling removal of heavy metals without requiring high operational pressures. Selectivity can be achieved through various means, including the use of specific organic chelating groups in polymer membranes, such as divinylbenzyl triethylenetetramine (diVB-TETA) for the specific removal of Cu (II) [213,214]. Alternatively, the introduction of high efficiency particle adsorbers into composite polymer-supported membranes is proving to be a flexible method to introduce additional functionality (e.g., producing stronger or fouling resistant composite films) as well as ion selectivity. Such an approach also enables much more practical utilisation of nanoparticles and other related nanomaterials [215].

It is beyond the scope of this review to give a comprehensive summary of relevant nanocomposite membranes for heavy metals removal, given the depth and breadth of current research in the area, although, recent relevant examples related to nuclear effluent treatment will be highlighted. An area that has gained particular recent interest in the nuclear industry (along with wider importance) is the use of graphene oxide (GO) based materials [216,217]. For instance, GO selective membranes have been altered via the capillary width to selectively uptake TcO_4^- ions, due to its low hydration free energy compared to other common ions such as Cl^-, SO_4^- and NO_3^-, although their efficacy has only been demonstrated for dilute anionic solutions [218]. Very recently Hu et al. [219], investigated modified GO membranes for the separation of radioactive caesium from cobalt, with separation factors >20 achieved. The inclusion of magnetic particles in membrane filters can additionally be used to enhance hydrophilicity and increase rejection of target species, such as evidenced by Abdi et al. [220], where copper removal and fouling reduction were achieved using a novel magnetic graphene oxide/metformin hybrid material.

There have also been various methods investigated to incorporate other nanoparticles with proven effectiveness for radioisotopes. As a singular example, here, we will focus on hexacyanoferrate precipitates and other Prussian Blue analogues, which are also used in present operations (i.e., at Fukushima) in a granular form where they are especially good at removing monovalent ions, such as caesium [221]. For instance, clay-hexacyanoferrate composite hydrogels have recently been proven to have excellent caesium absorption capacity and selectivity even at low caesium concentrations (0.2 ppm) in seawater over a wide pH range (2–12) as schematically presented in Fig. 11 [222]. Rheology testing revealed the composite hydrogel to be elastic under low strain and exhibited reversible, self-healing characteristic. This behaviour demonstrates the robustness of the fabricated hydrogel to possible damage during sample handling/operation. Prussian blue analogues have also been embedded into electrospun polymer nanofibers [223,224] or composite high porosity aerogels [225,226], although the latter would be applied more as monoliths than membranes.

5.2. *Magnetic nanoparticle adsorption-separation*

Magnetic nanoparticles are also of great interest for PI applications, as they provide a number of benefits; including the ease of separation through the application of a magnetic field [227], the adsorption and removal of specific heavy metals and radionuclides [228–232] and the potential to increase the efficiency or reduce fouling in other downstream unit operations, such as filtration and fluidised beds [233–235]. Although magnetic nanoparticles naturally have poor selectivity for adsorption of radionuclides, it is possible to modify the magnetic core to act as a protecting shell typically using coatings including SiO_2, TiO_2, mesoporous carbon, potassium zinc ferrocyanide, lead, chitosan, zeolite, graphene oxide and other forms of silica or by encapsulation in porous materials and polymer emulsions [230]. For instance, phosphate functionalised magnetic nanoparticles have been used for uranium adsorption [236], while polyethylenimine coated nanocomposites have been used to remove Cs-bearing clay particles in soil [231]. Magnetic nanoparticles have also recently been combined with Prussian Blue analogues (as discussed for use in membranes) to give enhanced Cs^+ removal and separation [237].

Certainly, the use of magnetic nanoparticles as intensified adsorption and separation systems have seen a rapid increase in research interest over the last decade. Research has been reported extensively in recent review papers [238,239] including specifically for their use in nuclear effluent treatment [230], where the reader is directed for more complete information. Despite the clear research interest, there are still operational barriers for the implementation of this technology in the nuclear industry. While a lot of progress has been made in the functionalisation of the magnetic particles, operational and capital cost are considered high, due to the novelty and complexity of the technology. Traditional magnetic separation also consumes high energy to generate an appropriate magnetic field. Additionally, due to their material cost, magnetic nanoparticles are often designed to be recovered and reused. Yet, it would be challenging to do so in the presence of the radioactive ions, limiting their economic use in nuclear applications.

Nevertheless, there have been recent advances in research directed to the efficient separation of magnetic nanoparticles and alternative ways of utilising magnetic species in composite systems, that goes some way to addressing these challenges. One of the most pertinent examples of magnetic separation being conducted at scale is the work of Namiki et al. [240], who used a traditional drum separation technique to separate radionuclide contaminated fly-ash slurry at scale (10 l sample). Investigations were focused on wastes that resulted from the Fukushima nuclear accident, where $> 99\%$ removal of caesium was achieved. Furthermore, researchers have studied the use of inline filter meshes to remove magnetic particles in pipe flow applications. Nakamura et al. [241] used magnetised mesh filters to remove caesium contaminated, magnetic polymer-zeolite composites, while more recently, Powell et al. [242] developed a magnetic tube mesh flow-through cell ('MagNERD') for >95% recovery of iron oxide particles, although, improvements in percentage recovery would be required for some nuclear applications.

The magnetic phase can also be adjusted to selectively adhere to target species to enable their separation using a magnetic field, permit magnetic field induced flocculation to entrap non-magnetic particles, allowing enhanced gravitational separation or drive fluidisation without particle mixing [234,243]. For example, recent work has shown the effectiveness of using a composite magnetic flocculant to aggregate and separate strontium contaminated soil through sedimentation [244] (see Fig. 12, a), while magnetic nanoparticles have also been embedded in functionalised polymer-clay composites for high caesium removal and facile recovery [245]. Furthermore, they may also allow for the magnetic force to enhance the performance of micro-fluidic beds, where strict product control or small process streams are necessary [246]. The inclusion of magnetic particles in membrane filters can additionally be used to reduce fouling or modify other membrane properties. In a study by Abdi et al. [220] increases in pure water flux, copper removal and fouling reduction were achieved by modification of a nonfilter membrane with a magnetic graphene oxide/metformin hybrid material. Additionally, multiple functionalised magnetic nanoparticles have been used in multi-step methods to remove Cs^+ from soils [247] (shown in Fig. 12, b), while the natural paramagnetic properties of certain clay types with high natural radioisotope uptake have also been utilised to recover high-dose soil samples at Fukashima, as a volume reduction method [248]. Thus, there are various application routes and scenarios to utilise magnetic separation in nuclear applications.

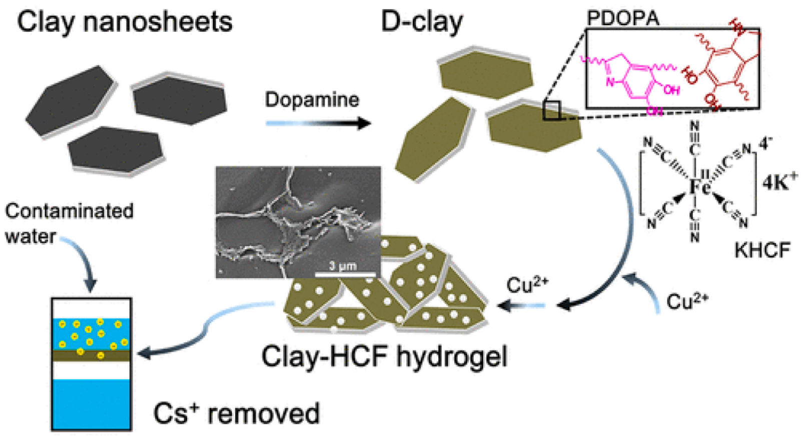

Fig. 11. Caesium removal in ultrapure water and seawater, using a doped nanoclay embedded membrane (reproduced from [222]).

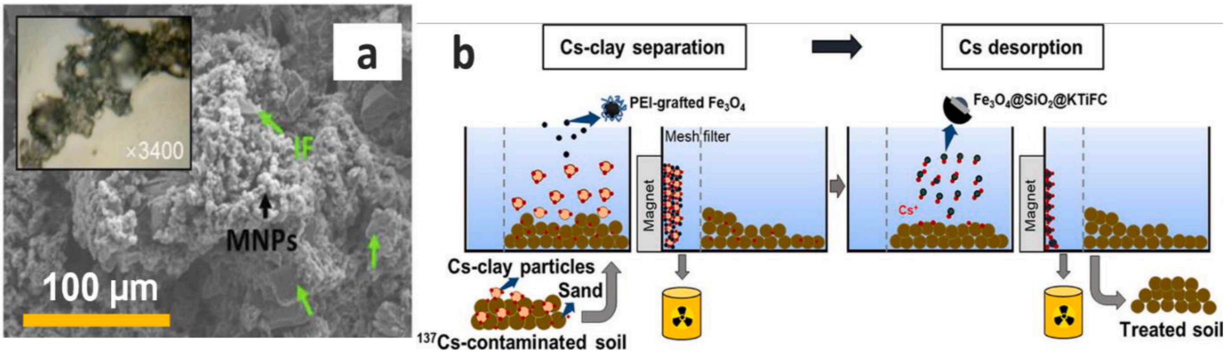

Fig. 12. a) FESEM image of a magnetic composite using magnetic nanoparticles (MNPs) and inorganic flocculant (reproduced and modified from [244]). b) Schematic of potential two-step process to remove Cs-bearing clay particles from soils, firstly using polyethylenimine (PEI)-grafted Fe_3O_4, and secondarily using composite KTiFC-functionalised magnetic silica particles (‘Fe_3O_4@SiO_2@KTiFC’) (reproduced from [247]).

6. Solid-liquid waste separators

6.1. Current utilisation of intensified designs

Solid-liquid separation and dewatering operations were one of the areas for early adoption, in what today would be classified as ‘intensified’ design options (although, in terms of effluent treatment, they have not been known specifically as such). However, due to the large process scales (especially to treat voluminous co-precipitation wastes) there has been a limit on the implementation of modern PI technologies, and gravitational separation is still widely practiced (e.g., through horizontal clarifiers [21]). Nevertheless, techniques such as ultrafiltration membrane systems are now widely used in dewatering, as discussed, and can be considered as an established intensified technique [249].

Another well-established intensification technique is the use of centrifugal forces, in this case to accelerate gravitational settling. Indeed, centrifugal separators have been used in wastewater treatment and minerals separation for many decades [250,251] while they are also established in the nuclear sector for waste treatment, as well as for the removal of solids from fuel dissolution operations, for instance [252, 253]. There has also been considerable progress in using centrifugation as a method to further enhance membrane filters [254] that has resulted in commercial products, such as the Spintek® intensified crossflow system (by Spintek Filtration Inc.). It has a series of circular, hollow porous plates made from sintered stainless steel, which are spun to increase shear forces across their surface, increasing typical fluxes to around ten times those seen in conventional crossflow filtration [252]. Downsides of such centrifugal designs are their increased mechanical complexity and wear over static separators, and the small relative flows achievable with high-speed systems.

There has also been development of intensified media filtration techniques, based on up-flow filter designs [255,256]. While traditional sand filtration is a cost-effective wastewater treatment process, it requires large operational areas from low process rates [257], as evidenced in the sand filtration tanks used at the SIXEP nuclear effluent treatment plant in Sellafield, U.K [156]. An example of a commercial intensified design is the self-washed sand filter, the Contiflow® by Huber Technology Ltd [258]. It operates on a continuous basis with backwashing occurring simultaneously with the filtration process, and therefore as other up-flow designs, no shutdown is required for backwash cycles. The Contiflow® has high operational reliability, low costs and requires minimum maintenance. The feed is introduced at the top of the filter and flows upwards through the bed, by way of a star shaped inlet distributor. The filter media is mobilised through an air-lift pump into the washer in the upper filter section, washing away the dirt, while allowing the heavier, coarser sand to fall back to the bed [258].

Additionally, there has recently been interest in the use of what is termed ‘mesofluidic’ separation, as an alternative to dead-end filtration [259,260]. Here, a series of inline flow-diversions or post arrays, lead to deterministic lateral displacement for particles of increasing size, allowing for continual size-based classification [261]. Interestingly, researchers at the Pacific Northwest National Laboratory (PNNL) in the US, have studied the use of large mesofluidic separator for the industrial scale diversion of particle fractions from Hanford waste streams, with efficiencies $>97\%$ being achieved [260].

6.2. Dissolved air flotation (DAF)

Apart from centrifugal separators, dissolved air flotation (DAF) may be considered one of the oldest intensified effluent treatment operations. While originally flotation was a method used to separate highly valuable minerals in mining and minerals processing, over the decades, DAF units have become common in industries such as water and industrial wastewater treatment as a rapid process to remove solids (as well as fats and grease). Essentially, the principle of gravitational sedimentation is turned on its head, and fine bubbles are introduced to adsorb particulate species, removing them in the froth phase. The separation of materials is mainly due to the physical chemistry of surface phenomena (surface interaction between the particle and microbubbles) and the hydrodynamics of the system. Generally, dissolved air flotation systems consist of 3 stages: a coagulation/flocculation stage, a pressurization tank/air injection system and a flotation cell [262,263]. Alternative dispersed air floatation designs, such as the Jameson cell, are more extensively applied in minerals processing [264].

While PI terminology is not always associated with flotation techniques, it is a process that can lead to considerable footprint reduction and increased throughput rate, in comparison to gravitational separators. Yet, unlike centrifuges, there has been no major uptake of DAF technologies in the nuclear reprocessing or waste sectors. Despite this, there is now considerable research interest in using flotation as a rapid separation and dewatering technique, especially for nuclear effluent treatment applications. Research has investigated the use of flotation to rapidly separate coprecipitate wastes bearing heavy metals [265–267], radioisotope-bearing composite clays and ion exchangers [268–270], solids waste management [271,272] and even direct solution removal of caesium and other heavy metals using ion foam flotation [273–276].

As an example, Rashad *et al.* [265] demonstrated a two-step process for removing ^{137}Cs, through initial co-precipitation with cadmium ferrocyanide and secondary flotation with surfactant collectors. Similarly, Wu and co-workers [267] investigated the flotation of humic acid modified hydroxide precipitates for decontamination of a variety of divalent heavy metals. Recently, Yusuf Prajitno *et al.* [268] demonstrated high dewatering ratios, for the separation of caesium contaminated clinoptilolite floated using cationic surfactants as collectors, while Lockwood et al. [271], produced similar performance for the flotation of magnesium hydroxide sludges from corroded nuclear fuel cladding.

Ortiz-Oliveros and Flores-Espinosa [262] investigated the removal of oil and ^{60}Co with DAF using a lamella module, achieving over 80% removal of non-radioactive cobalt at an initial concentration of 6.5 mg/l in 8–10 min. The same authors have since designed and tested a mobile DAF unit with a volume of 0.15 m^3 as a response to on-site nuclear plant or environmental emergencies, with total Co and ^{60}Co removal levels of ~94% and 75% respectively [263] (cell schematically shown in Fig. 13). Mobile treatments systems offer greater flexibility compared to permanent facilities. In case of emergencies, wastes need to be managed at the site of operation and in the smallest time frame possible. Therefore, DAF units offer modular designs that could be incorporated both in normal operations and for critical waste clean-up activities.

6.3. Perspectives and future directions

Within this review (and in the summary Table S1 presented within the ESM) our perspective has been to consider what PI technologies have realistic potential for transfer to nuclear effluent operations within the next 10–15 years, and what the current barriers to implementation are. For example, a multitude of intensified plug flow reactors were outlined that varied greatly in their nuclear applicability. Designs ranged from microreactors that offer fine control over operating conditions, but low technology transfer probability (due concerns in channel clogging and scale-up) to much larger designs, including oscillatory baffle and agitated tubular reactors. Generally, systems that intensify by more passive means are less space efficient than mechanically complex options, presenting one of the critical challenges in balancing the needs of the nuclear industry. In terms of adsorbers or ion exchangers, there were less mechanically active intensified units, with the majority of research on centrifugal systems, like rotating packed beds (RPBs). Conversely, there were a number of non-mechanical options with relatively high technology transfer capability also investigated, such as the use of additively manufactured monoliths. These kinds of exchange materials could also be incorporated into tubular adsorbers, (e.g., agitated tubular reactors) providing combined modular solutions that are cost effective to manufacture. For waste separation, recent research has focused on both possible step-change processes that still require considerable industrial development (including chemically functional ultrafiltration membranes and magnetic nanoparticles) as well as industrially established, scalable techniques, such as dissolved air flotation (DAF).

A number of themes are apparent when considering the further challenges towards implementation. It is firstly clear that despite a lot of research promise, and reductions in manufacturing costs, there still isn't many relevant examples of modular treatment technologies being scaled-out to levels relevant for effluent treatment, especially regarding reactors. While some reactor designs (e.g., ATR, OBR) offer more flexible approaches, a number of units would still be needed to achieve the required 10 s to 100 s m^3/day capacity, potentially limiting any perceived gains in efficiency. As a lot of more passive PI designs require scale-out to much greater extents (e.g., TCRs) obstructions to implementation may be even more considerable. To more fully understand the technoeconomic advantages or costs of different designs, it is suggested that broader multicriteria optioneering studies are completed, to compliment the technical review herein.

Also, the mechanical complexity of some of the designs is a particular concern in high radiation environments, where worker protection is a paramount concern. The use of rotating, shaking or centrifugal designs have to be considered in terms of the additional wear and failure rates, as operational downtime in high radiation environments have much greater economic and safety implications than other areas. Nevertheless, we don't believe such mechanical challenges are insurmountable. Indeed, given that centrifugal contactors and separators are already being applied in fuel reprocessing environments, it is clear there are examples of mechanically complex designs being built to extremely high operational and safety tolerances.

Another interesting consideration is the latent differences to physicochemical conditions of processed wastes with PI solutions, and prospective impact on downstream processing. For example, most intensified plugflow reactors for co-precipitation generally result in finer crystals being produced due to enhanced shear environments (as well as the benefit of being more monodispersed). As such, there is some potential for wastes to be more difficult to separate, although, intensified separation methods like DAF, do not have the same issues as traditional gravitation separation. There is also a clear need to consider the entire waste cycle more holistically, to ultimately allow the integration of waste treatment processes with downstream encapsulation or other solidification abatement techniques. For instance, the use of 3D structured monoliths, magnetic nanoparticles or embedded membranes may provide exciting avenues for intensified exchange but may also present difficulties in ultimate disposal. However, it has to be noted that similar issues exist with current treatment techniques, with considerable compatibility issues between wastes and different solid encapsulation materials. Indeed, a move towards PI designs may be an advantage, as both material and process can be developed collectively to ensure optimal long-term solutions. Regardless of these challenges, there is no doubt that nuclear fission needs to overcome its *waste* problem to be truly considered a viable 21st century technique for low-carbon power, and PI offers an exciting route towards industrial sustainability.

7. Conclusions

In this state-of-the-art review, technologies and techniques related to the process intensification (PI) of nuclear effluent treatment operations have been described. Our main focus was on highlighting alternatives to current industry standard continuous stirred tank reactors (CSTRs) and column contactors, which may provide step-change improvements to space and time efficiency or increases in modularity. As such, the review considered separate options for precipitators and reactors, contactors

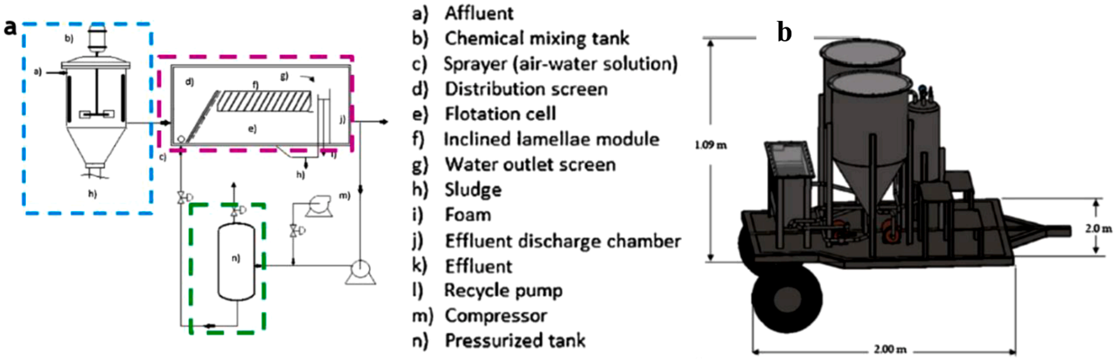

Fig. 13. a) Mobile dissolved air flotation (DAF) system for the treatment of liquid radioactive waste. Dashed box delineates the flocculation stage (blue), pressurization tank (green) and flotation cell (purple) and b) 3D render of the same unit with dimensions (reproduced and modified from [263]).

and ion exchange systems, as well as downstream solid-liquid waste separators, where a number of units identified could act in a combination of ways. Some of the difficulties highlighted in technology transfer of PI to effluent treatment, include the relatively large scale of operations with regards to current areas where designs are applied (e.g., high value chemicals). Also, the requirement for reduced maintenance in nuclear process plants to mitigate risk also limits some intensified options, which can have increased mechanical complexity. In general, there is a trade-off between the operational simplicity available from smaller plug-flow units, and their greater difficulties in scaling-up to appropriate levels for effluent treatment.

Despite these restrictions, several technologies exist for the intensification of nuclear effluent treatment that have been demonstrated at scale and are mature, and indeed, this review highlights that a significant amount of recent research exists for intensified designs that are already being applied to nuclear waste systems and related areas (e.g., fuel reprocessing). Looking forward in terms of nuclear waste management in particular, there are several avenues of opportunities. A more robust industrial approach is required to assess technologies, based on key performance indicators for specific nuclear utilisation, rather than their inherent characteristics. Also, process integration, which is slightly discussed in this paper by considering combined precipitators/adsorbers and combined adsorbers/separators, should be taken to a strategic level in a holistic and circular approach to processes of concern. Nevertheless, findings from the review demonstrate that the next generation of nuclear operations should adopt the principles of PI in effluent treatment flowsheets, as this can provide significant economic and safety benefits, while existing technical barriers are no longer insurmountable.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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Supplementary materials

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