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THE HIDDEN FOOTPRINT OF HYDROGEL ADSORBENTS: RESIDUAL MONOMER, CROSSLINKER AND NANOFILLER LEACHING AND THE ECOTOXICITY OF TREATED WATER – A CRITICAL REVIEW

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ABSTRACT

Hydrogel adsorbents are currently among the most investigated materials for the removal of colours, metals and medicines from water. Most articles evaluate them by capacity, speed and number of reusecycles . Hardly anybody asks what the gel leaves in the water that it has just cleansed. This review tackles that hidden footprint. We collect the evidence for three kinds of leachable species: leftover monomers such as acrylamide and acrylic acid, unreacted crosslinkers with initiator residues and nanofillers that shed particles or ions, principally Ag, ZnO, Fe3O4 and graphene oxide. Release routes are traced over the whole service life, from initial wash after manufacture to regeneration in acid or base and eventually disposal. Analytical approaches for the detection of these species at regulatory levels are discussed; acrylamide can be detected at low ng L−1 by LC-MS/MS and dissolved and particulate metal are separated by ICP-MS after ultrafiltration. The toxicity data are grossly inconsistent. Acute effects of acrylamide on algae, daphnids and fish occur only at mg L−1, although drinking-water limitations are 0.1–0.5 µg L−1 and dissolved silver is active below 1 µg L−1. However, most research on the environmental safety of hydrogels are based on a single seed germination test. We suggest a tiered approach combining leachate chemistry with multi-trophic bioassays and safe-by-design steps: cleaner starting and cross-linking chemistry, confirmed purification and solid anchoring of nanofillers. Proposed minimal reporting standard will verify claims of clean water.

Keywords: *Hydrogel adsorbent; Residual acrylamide; Crosslinker leaching; Nanofiller release; Ecotoxicity; Safe-by-design.*

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1. INTRODUCTION

Adsorption is cheap, simple and works for many pollutants. Over the past decade hydrogels have moved to the centre of this field. Their swollen networks carry many functional groups, they can be loaded with nanoparticles, and they are easy to shape into beads, films or columns [1,2]. The literature now holds hundreds of grafted, composite and magnetic gels aimed at dyes, metals and drugs. The usual report card is familiar. It gives the maximum capacity from a Langmuir fit, the kinetic model that fits best, and the removal left after four or five cycles. What it rarely gives is the make-up of the water after treatment, apart from the target pollutant. This is a real blind spot. Many adsorbent gels are built from acrylamide, a neurotoxic monomer that IARC classes as probably carcinogenic to humans [3]. Others are crosslinked with glutaraldehyde or N,N′-methylenebisacrylamide, both on the list of synthetic crosslinkers regarded as harmful to people and the environment [4]. Many also carry silver, zinc oxide, magnetite or graphene oxide, and dispersed nanomaterials can escape into the treated stream [5,6]. Even the polymer backbone may persist. Acrylate superabsorbents have been described as a new kind of microplastic [7,8]. So the question is a simple one. Does a hydrogel that removes a dye also add something to the water? Reviews on the management of spent adsorbents have pointed out that emerging adsorbents seldom come with any analysis of their environmental impact [9,10]. As far as we know, no review has yet gathered the leaching and ecotoxicity evidence for hydrogel adsorbents in one place. This article tries to do that. We first map the leachable species and the ways they are released, then the analytical tools, then the toxicity data, and finally the design and reporting steps that could close the gap.

2. WHAT CAN LEACH FROM A HYDROGEL ADSORBENT

A swollen gel is not one clean network. It holds a sol fraction of chains that never joined the network, small molecules left over from synthesis and, in composites, inorganic particles held by fairly weak forces. Figure 1 sketches this inventory. Table 1 lists the common building blocks and why each one matters.

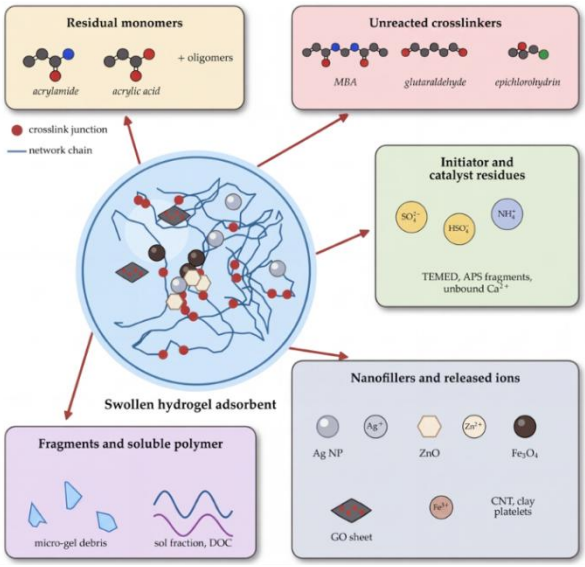


Figure 01: Leachable inventory of a typical hydrogel adsorbent: residual monomers, unreacted crosslinkers, initiator and catalyst residues, nanofillers with their released ions, and gel fragments with soluble polymer.

Table 01: Common hydrogel building blocks, the species they can release, and why these species matter.

Component	Role in the gel	Possible leachate	Concern or benchmark	Ref.
Acrylamide (AAm)	Monomer	Free AAm, oligomers	Neurotoxic; IARC Group 2A; drinking-water limits 0.10 µg L^{-1} (EU) and 0.5 µg L^{-1} (WHO)	[3,11,12]
Acrylic acid and acrylates	Monomer	Free AA, short PAA chains	Low acute toxicity; chelation of nutrients in soft water; not biodegradable	[13,14]
N,N′-Methylene-bisacrylamide (MBA)	Crosslinker	Unreacted MBA, hydrolysis products	Listed among harmful synthetic crosslinkers; amide links hydrolyse in strong base	[4,15]
Glutaraldehyde (GA)	Crosslinker for chitosan, PVA, gelatin	Free aldehyde	Dose-dependent cytotoxicity of extracts; toxic in Microtox at high loading	[16–18]
Epichlorohydrin (ECH)	Crosslinker for polysaccharides, cyclodextrins	Free ECH	Regulated in drinking water (0.10 µg L^{-1} in the EU)	[4,11]
APS, KPS, TEMED	Initiator, accelerator	Sulfate, amine residues	Choice of initiating system controls residual monomer	[19,20]
Ag nanoparticles	Antibacterial filler	Ag^{+}, particles	*D. magna* EC_{50} 0.75 µg L^{-1} as total Ag	[21,22]
ZnO, CuO	Photocatalytic or antibacterial filler	Zn^{2+}, Cu^{2+}, particles	ZnO median algal $L(E)C_{50}$ 0.08 mg L^{-1}; faster dissolution at low pH	[22,23]
Fe_3O_4	Magnetic separation	Fe ions, particles	Leaching and aggregation over reuse	[24]

			cycles	
GO, CNT	Reinforcement, adsorption sites	Sheets, fragments	GO PNEC 20–100 µg L^{-1}; 5–23 µg L^{-1} with humic acid	[6,25]
Chitosan, alginate	Biopolymer backbone	Soluble chains, DOC, Ca^{2+}	Low-molecular-weight chitosan EC_{50} about 12 mg L^{-1} (crustaceans)	[26]

2.1. Residual monomers and oligomers

Free-radical polymerization rarely runs to full conversion inside a gel. As the network forms, viscosity climbs and the last monomer molecules get trapped. Kabiri and co-workers showed that the initiating system alone changes the residual monomer of acrylic-acid superabsorbents, and that a two-step initiation can lower it further [19,27]. Commercial products are not free of the problem. Yılmaz et al. found acrylamide in all five baby-diaper superabsorbents they tested, at 0.483–1.36 µg g^{-1} [28]. In agricultural poly(acrylamide-co-potassium acrylate) gels, the residual acrylamide responded to temperature, salinity and time [29,30].

These figures look small. But a gel dosed at 1 g L^{-1} that releases only 1 µg g^{-1} of acrylamide would add about 1 µg L^{-1} to the water. That is ten times the EU parametric value of 0.10 µg L^{-1} and twice the WHO guideline value of 0.5 µg L^{-1} [11,12]. The US EPA controls acrylamide through a treatment technique instead, allowing polymer with no more than 0.05% monomer dosed at 1 mg L^{-1} [31]. Adsorbent doses are often a thousand times higher than flocculant doses. The arithmetic alone should make residual monomer a routine item in adsorbent papers.

Field data confirm that the use of polyacrylamide leaves measurable monomer behind. Acrylamide reached 280 ng L^{-1} at a sand-and-gravel mine [32] and ranged from 5.2 to 35.7 ng L^{-1} in Romanian drinking water [33]. Guezennec et al. reviewed how polyacrylamide flocculants move and degrade in hydrosystems, and stressed that they may carry residual monomer with them [34]. Acrylic acid draws less attention because it is less toxic. Even so, residues in commercial superabsorbents have been blamed for poor germination and crop growth, and a modified synthesis cut residual acrylic acid to 0.03% [35].

2.2. Crosslinkers and initiator residues

Crosslinkers are used in small amounts, so they are easy to forget. Most are more reactive, and more toxic per molecule, than the monomer. Glutaraldehyde is the classic case. Unreacted aldehyde can diffuse out of chitosan materials and harm cells [18]. Extracts of glutaraldehyde-crosslinked chitosan left only 41% of fibroblasts viable, while a gel crosslinked by gold nanoparticles showed no cytotoxicity [16]. Gelatin/chitosan microspheres made with 2.5 wt% glutaraldehyde were cytotoxic, whereas those made with 0.5–1 wt% were not [17]. Retinal cells exposed to glutaraldehyde-treated chitosan also showed more apoptosis than cells exposed to genipin-treated material [36].

N,N′-Methylenebisacrylamide (MBA) is the default crosslinker for acrylamide and acrylic gels. Apart from its own toxicity, it has a weak point that matters for regeneration. Its amide links hydrolyse in strong alkali. MBA began to decompose in 1 M KOH after several days, and strongly alkaline gels crosslinked with it turned liquid after 28 days of ageing at 60 °C [15]. Repeated washing with NaOH between cycles is therefore not a neutral step. Epichlorohydrin, common in polysaccharide and cyclodextrin networks, is regulated as a drinking-water contaminant in its own right [11,31].

Initiators leave residues too. Persulfate gives sulfate and bisulfate, and TEMED or other amine accelerators remain unless they are washed out. Xiang et al. noted that both residual acrylamide and some initiators limit the safe use of polyacrylamide gels; they replaced ammonium persulfate with gallium nanoparticles or MoS_2 nanosheets to lower the monomer left behind [20].

2.3. Nanofillers and their ions

Nanocomposite gels are attractive because the filler adds surface area, magnetism, catalytic activity or antibacterial action [1,2]. The same filler is the most likely source of secondary pollution. Yu et al. designed tough, self-healing gels precisely because dispersed nanomaterials flow so easily into the water stream [5]. For graphene oxide held in chitosan, Alves et al. noted that the sheets can leach during adsorption, and called for studies that measure this release [6].

Measured release is not trivial. A PEGDA/gelatin gel made with silver from 200 ppm $AgNO_3$ released 0.35 ppm of silver in seven days [37]. Silver–chitosan nanocomposites shed about 1% of their silver as ions, and that fraction tracked their toxicity [26]. Zinc oxide is easily soluble. In cellulose acetate composites, the zinc released in the dark increased with the ZnO loading [38]. ZnO dissolves more rapidly at low pH [23]. Magnetic adsorbents are susceptible to performance loss in cycling due to aggregation, leaching and surface passivation [24]. CuO-loaded ultrafiltration membranes shed particles after extensive exposure to cleaning solutions and the authors recommended a polishing step downstream [39]. Membranes are not gels, of course, but the lesson is the same.

3. RELEASE ROUTES OVER THE SERVICE LIFE

Leaching is not a single event. It follows the life of the adsorbent, as Figure 2 shows, and four stages deserve attention.

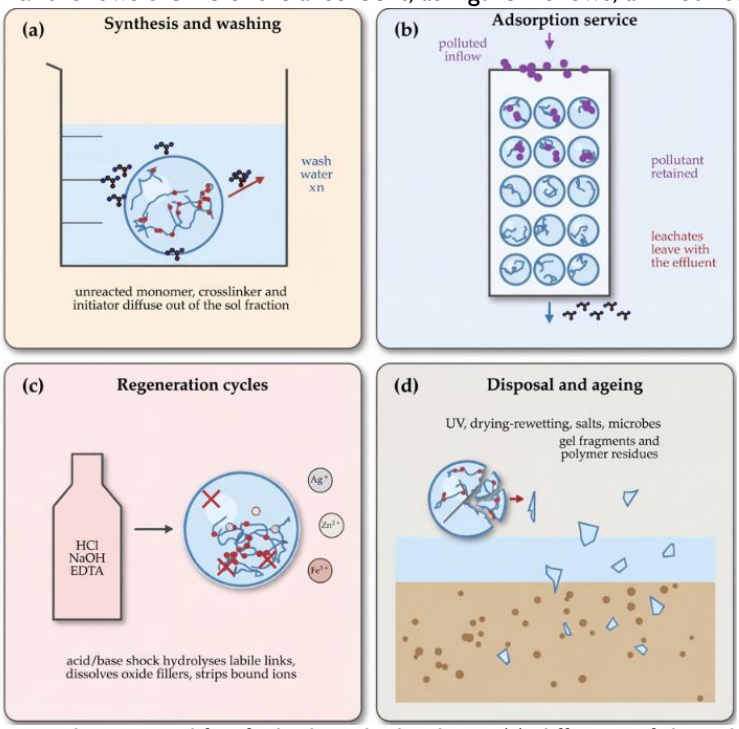


Figure 02: Release routes over the service life of a hydrogel adsorbent: (a) diffusion of the sol fraction during washing, (b) leachates leaving with the effluent during adsorption, (c) acid or base shock during regeneration, and (d) fragmentation and ageing after disposal.

3.1. Washing after synthesis

The first contact with water removes most of the sol fraction. Diffusion controls this step, so release is fast at first and then tails off. Brynda et al. washed poly(HEMA) gels and found that most impurities left within hours, yet one unidentified compound was still eluting after several months [40]. Zinc migration from nanocellulose/ZnO films also followed Fickian diffusion, with diffusion coefficients of 10^{-16}–10^{-15} m^2 s^{-1} [41]. Slow tails like these mean that a gel which passes a quick wash test can still bleed during months of service.

3.2. Adsorption service

The gel is used in a genuine water matrix. Swelling and release are affected by ionic strength, hardness and natural organic matter. Silver release from alginate encapsulated nanoparticles decreased from 19.3 µg L−1 in ultrapure water to 0.83 µg L−1 in hard water [42]. The release from commercial nano-enabled items also depends on the receiving water [43]. Hence, testing performed in deionised water solely may over- or underestimate what occurs in a plant. Ion exchange is also important. Pollutant cations can replace ions trapped in the gel, such as Ca2+ in alginate, and push them into the water.

Physical form adds another layer. Grinding a bulk gel into powder speeds up swelling and uptake [44]. The larger surface that helps adsorption will, by the same token, speed up release, a trade-off that is rarely discussed. In fixed beds the first bed volumes usually carry the heaviest load of leachate. A short flush to waste at start-up is cheap insurance, but it only works if someone has measured how long the flush should be.

3.3. Regeneration

Regeneration is probably the harshest stage. Hydrochloric acid and sodium hydroxide are the most used eluents for metals and anionic dyes [45,46]. Acid dissolves oxide fillers and strips metal ions, while base hydrolyses amide and ester links. We have already seen that MBA crosslinks fail in strong alkali [15]. In alginate-immobilized Cu–Fe catalyst beads, the loss of carboxyl groups pointed to depolymerization of the alginate, even though metal leaching was lower than from the bare catalyst [47]. Each cycle can thus produce a pulse of leachate. Reuse studies, however, usually report only the removal percentage, and not what came out in the eluent or in the first effluent after regeneration.

3.4. Disposal and ageing

At the end of its life the spent gel is landfilled, burned or, in some proposals, spread on soil. Acrylate superabsorbents are not readily biodegradable [7]. Under weathering they may form plastic-like solid residues in soil [8], and their properties shift with soil solutes and drying–rewetting cycles [48]. A dye-loaded gel adds the desorbable pollutant to this picture. Options for managing spent adsorbents have been reviewed in detail [10,49], but leaching tests on spent hydrogels remain rare.

4. ANALYTICAL TOOLS FOR LEACHATES

One reason leaching is under-reported is that it is hard to measure. Acrylamide is small, very polar and nearly invisible in the useful UV range. Metals must be split into dissolved and particulate forms. Graphene oxide has no simple elemental tag. Table 02 summarizes methods that have worked in water and in polymer extracts.

Table 02: Analytical methods reported for hydrogel leachables and related species in water.

Analyte	Method	Matrix	Performance	Ref.
Acrylamide	SPE on activated carbon + LC-MS/MS, $^{13}C_3$ internal standard	Drinking water	LOD 0.02 µg L^{-1}; LOQ 0.1 µg L^{-1}	[50]
Acrylamide	Large-volume injection HILIC-MS/MS	River and drinking water	MDL 2.4 ng L^{-1}	[32]
Acrylamide	Coevaporation + isotope-dilution LC-APCI-MS/MS	River water, field runoff	LOD 0.02 ng mL^{-1}; LOQ 0.06 ng mL^{-1}	[51]
Acrylamide	Offline SPE-LC-MS	Drinking water	LOQ 2 ng L^{-1}	[33]
Acrylamide	Bromination + DI-SPME-GC-MS	Drinking water	LOD 0.05 µg L^{-1}	[52]
Acrylamide	Nonaqueous CE with sample stacking	Superabsorbent extracts	LOD 5.3 µg L^{-1}; LOQ 17.7 µg L^{-1}	[28]
Acrylamide	HPLC-UV after SPE	Medical polyacrylamide gel	Residual monomer in the gel (n.s.)	[53]
Unreacted monomers	^{1}H NMR after dialysis	Polymer solutions	Removal up to 99% confirmed	[54]
Ag, dissolved and total	0.22 µm filtration + ICP-OES	Hydrogel leachate	Release followed at 24 and 96 h	[55]
Ag speciation	Ultracentrifugation and membrane filtration; GF-AAS, ICP-OES/MS	Ecotoxicity test media	Dissolved Ag predicted and validated	[56]
Ag	ICP-MS, EDXRF	Leachates in waters of different hardness	0.83–19.3 µg L^{-1} measured	[42]
CuO nanoparticles	DLS, EDS, ICP-MS	Membrane cleaning solutions	Leaching confirmed after 840 h	[39]
Zn^{2+} and ZnO particles	Multi-technique analysis with Fick and Weibull models	Water, 10% ethanol	D = 10^{-16}–10^{-15} m^2 s^{-1}	[41]

For acrylamide, LC-MS/MS with isotope dilution is the reference approach. Solid-phase extraction on activated carbon gave a quantification limit of 0.1 µg L^{-1} in tap water [50]. Large-volume injection with hydrophilic-interaction chromatography reached a detection limit of 2.4 ng L^{-1} after a simple solvent exchange [32], and offline SPE-LC-MS pushed quantification down to 2 ng L^{-1} [33]. Coevaporation before LC-APCI-MS/MS is another route for river water [51]. Where LC-MS/MS is not available, GC-MS after bromination is a good alternative [52]. Gel extracts carry higher levels, so HPLC-UV with clean-up [53] or capillary electrophoresis with sample stacking [28] is usually enough. Simple 1H NMR can demonstrate the disappearance of the vinyl signals of the unreacted monomer after dialysis [54,57].

Separation is the crucial stage for metal fillings. Dissolved and particulate silver are separated by filtration at 0.22 µm followed by analysis by ICP-OES [55] or ultracentrifugation and membrane filtration prior to ICP-MS [56]. Köser et al. showed that the toxicity of a reference silver nanomaterial was determined by the Ag+ existing in the stock dispersion and not by later oxidation [56]. DLS and EDS were used to confirm the departure of particles, not only ions, from the matrix [39].

Here a generic caution is applicable. The gel fraction is the most typical purity number in hydrogel publications and does not tell us anything about the amount of mobile material. It quantifies the remainder. Leachate must be measured directly in the water matrix of use at a defined solid to liquid ratio, time and temperature.

The weak point still remains graphene oxide and gel fragments. There is no standard way for counting GO sheets at µg L−1 in an actual effluent, which is one reason why release from GO/chitosan gels is so poorly described [6]. Total or dissolved organic carbon provides a lumped signal for soluble chains and fragments, effective for screening but blind to identity. Buchmann et al. assessed the qualitative and quantitative approaches available today for solid superabsorbent residues and recommended for comprehensive monitoring measures [8]. Until better techniques are available, an acceptable minimum is mass balance before and after use and particle counts in the effluent.

5. ECOTOXICITY: WHAT THE DATA SAY

Figure 3 places the available toxicity values for the main leachables on one log scale, next to drinking-water limits. The values span six orders of magnitude, and their order is telling.

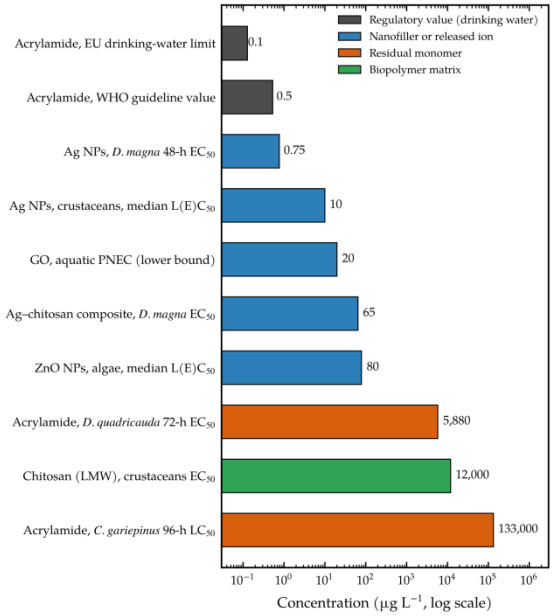


Figure 03: Toxicity and regulatory benchmarks for hydrogel leachables on a common scale (µg L^{-1}). Sources: EU limit [11]; WHO guideline [12]; Ag NP EC_{50} for *D. magna* [21]; median $L(E)C_{50}$ of Ag NPs for crustaceans and of ZnO NPs for algae [22]; GO PNEC [25]; Ag–chitosan composite and low-molecular-weight chitosan EC_{50} [26]; acrylamide EC_{50} for *D. quadricauda* [58]; acrylamide LC_{50} for *C. gariepinus* [59].

5.1. Monomers and soluble polymer

Acrylamide exhibits a slight acute effect on aquatic species, while early life stages are the most vulnerable [60]. Algae were the most sensitive group in a conventional test battery, and Daphnia magna and Lemna minor evaluated it as somewhat hazardous [61]. The EC50 for Desmodesmusquadricauda after 72 h was 5.88 mg L−1 and visible alterations in cell morphology were observed [58]. The 96-h LC50 for African catfish was 133 mg L−1 while sublethal exposure triggered oxidative damage and DNA breakage [59]. Zebrafish embryos exposed to 10–300 mg L−1 revealed abnormalities and reduced swimming performance [62]. Acute toxicity to zebrafish embryos varied by two orders of magnitude (0.16–33 mM) for 10 acrylamide-type monomers and followed hydrophobicity and reactivity [63].

The practical point is an uncomfortable one. The drinking water restrictions for acrylamide are based on carcinogenicity and neurotoxicity in mammals and not on acute ecotoxicity. effluent treated in a processpass every standard bioassay and still exceed the EU limit a thousand-fold. For this monomer, bioassays cannot replace chemical analysis.

Soluble polyacrylates behave differently. They are generally of low toxicity, and the effects seen on algae and crustaceans in soft water are put down to chelation of nutrient cations [13]. Still, a liquid polyacrylic-acid polymer inhibited *Aliivibrio fischeri* luminescence by 73% and nitrifying bacteria by 88% at 100 mg L^{-1}, and none of the polymers in that study were biodegradable [14]. A potassium polyacrylate superabsorbent also reduced the biomass of *Chlorella vulgaris*, mainly at high cell density [64].

5.2. Crosslinkers

Toxicity data for crosslinkers come mostly from biomedical work, and hence from mammalian cells. Aquatic data are scarce. The few Microtox results are instructive, though. Chitosan films with 15% glutaraldehyde left 24% of *A. fischeri* cells viable, whereas films crosslinked with dialdehyde chitosan were less toxic [18]. In the same work uncrosslinked chitosan lowered bacterial viability by 61%. This is a reminder that an antibacterial matrix will score as toxic in any bacterial test. Dialdehyde polysaccharides gave hydrogels with good cyto- and hemocompatibility, while glutaraldehyde and genipin both showed marked cytotoxicity under the same conditions [65]. A natural origin, then, does not guarantee a harmless crosslinker.

5.3. Nanofillers and released ions

Silver is the most sensitive case. For powder-type silver nanoparticles the 48-h EC_{50} for *D. magna* was 0.75 µg L^{-1} as total silver and 0.37 µg L^{-1} as dissolved silver [21]. In a survey of 317 values, the median $L(E)C_{50}$ of Ag nanoparticles for crustaceans was 0.01 mg L^{-1}, and that of ZnO nanoparticles for algae was 0.08 mg L^{-1} [22]. Embedding silver in a polymer softens the effect but does not remove it. Silver–chitosan nanocomposites still gave EC_{50} values of 0.065–0.232 mg L^{-1} for *D. magna* and were ranked as very to extremely toxic [26]. A TEMPO-cellulose silver hydrogel was about ten times less toxic to zebrafish embryos than free Ag^{+}, with LC_{50} values of 1.115 and 0.1228 µM, respectively [55]. Silver encapsulated in alginate showed the most harmful leachates to daphnids and sea-urchin larvae. The authors cautioned that the alginate may dissolve and remobilise silver with time. Treated encapsulation was viewed as a means to modulate exposure rather than to remove the hazard [42].

Graphene oxide is less frightening based on current evidence. A species-sensitivity method resulted in projected no-effect doses of 20–100 µg L−1, declining to 5–23 µg L−1 in the presence of humic acid [25]. Effects were concentration dependent in chronic studies at three trophic levels, however the risk was low at environmentally relevant exposure.[66]. The humic-acid result deserves attention, since natural waters always carry organic matter.

The biopolymer matrix is not inert either. Low-molecular-weight chitosan was harmful to crustaceans at about 12 mg L^{-1} [26]. Soluble chains and fragments from chitosan gels could therefore add to the toxicity of an effluent, especially in bacterial tests.

5.4. How treated water is being tested

Against this background, current practice in hydrogel papers is thin. When a safety test is included at all, it is usually seed germination. A lignin/dimethylacrylamide gel was rated non-phytotoxic to maize [67]. Water treated with a Nyssa gum–poly(acrylic acid) gel gave 100% germination, the same as distilled water [68]. An alginate–cactus mucilage gel showed no toxicity to lettuce seeds [69], and a modified guar gum lowered dye toxicity to mung beans [70]. These results are useful, but limited. Germination is a coarse endpoint, seeds tolerate many dissolved chemicals, and the comparison is often made against the dye solution rather than clean water. Such a test shows that the dye was removed. It does not show that nothing was added. Gels built from acrylamide and acrylic acid are also proposed for water reuse [71]. This makes the gap more serious.

There are better models in the related areas. New phosphorus adsorbents were tested in algal growth and D. magna experiments with direct exposure to the material and indirect exposure via the surrounding water, which helps to differentiate particulate effects from dissolved ones [72]. The activated biochar employed for the removal of pharmaceuticals was assessed by the ecotoxicity of the treated water itself [73]. ZnO–CuO photocatalysts were tested for simultaneous removal efficiency and multi-trophic toxicity [74]. Physicochemical data alone could not be shown to decrease acute or chronic toxicity in landfill-leachate treatment [75]. The selection of organisms for effluent testing has been widely addressed [76]. Table 3 summarises research that investigated leaching or toxicity for hydrogel and related adsorbent materials. A tiered structure based on these studies is proposed in Figure 4.

5.5. Mixtures, carriers and transformation

Treated water is never a solution of one chemical. It has the leachates, the pollution that was not eliminated, and whatever the two make together. Nanofillers make this more complicated. Graphene oxide can transmit polycyclic aromatic hydrocarbons into aquatic organisms [6]. The presence of humic acid lowered the expected no-effect level of graphene oxide [25]. And transformation doesn't always mean cleansing, either. The silver sulphide, typically considered as an innocuous substance, was made lethal in the acidic gut cavity of Hydra vulgaris [77]. Testing leachate and treated effluent as entire mixtures, instead of one component at a time, captures these interactions without the need to forecast them.

Table 03: Previous studies that measured leaching or toxicity of hydrogel and related adsorbent materials.

Material	What was measured	Test system	Main outcome	Ref.
Commercial polyacrylamide-based superabsorbents (diapers)	Residual acrylamide	Nonaqueous CE	0.483–1.36 µg g^{-1} in all five samples	[28]
Poly(AAm-co-K acrylate) agricultural gels	Residual AAm under heat, salinity and time	Chemical analysis	Residual AAm responded to abiotic stress and ageing	[29,30]
Chitosan films crosslinked with GA, DAS or DACS	Acute toxicity of films	Microtox (*A. fischeri*)	15% GA left 24% viability; DACS less toxic	[18]
Chitosan hydrogel crosslinked with GA or Au nanoparticles	Extract cytotoxicity	Mouse embryonic fibroblasts	GA extract 41% viability; Au-crosslinked gel non-cytotoxic	[16]
PEGDA/gelatin–AgNP hydrogel	Silver release	Chemical analysis	0.35 ppm Ag released in 7 days	[37]
Silver–chitosan nanocomposites	Ag^+ shedding, acute toxicity	*D. magna*, *T. platyurus*, *V. fischeri*	*D. magna* EC_{50} 0.065–0.232 mg L^{-1}; about 1% of Ag shed as ions	[26]
Alginate-encapsulated mycogenicAgNPs	Ag release, leachate toxicity	*D. magna*, *P. lividus*, *D. rerio*, *R. subcapitata*	Release 19.3 µg L^{-1} (ultrapure) to 0.83 µg L^{-1} (hard water); *D. magna* LOEC 0.781% leachate	[42]
TEMPO-cellulose–AgNP hydrogel	Ag leaching, embryo toxicity	Zebrafish FET	LC_{50} 1.115 µM vs 0.1228 µM for $AgNO_3$	[55]
Cu–Fe catalyst in alginate beads	Metal leaching	AAS, FTIR	Lower leaching than bare catalyst; alginate partly depolymerized	[47]
Polyacrylic-acid water-	Ecotoxicity,	*L. minor*, *P.*	Low to moderate effects; 73%	[14]

soluble polymers	biodegradability	*subcapitata*, *D. magna*, *A. fischeri*, activated sludge	luminescence and 88% nitrification inhibition at 100 mg L^{-1}	
Novel phosphorus adsorbents	Direct and indirect exposure	*R. subcapitata*, *D. magna*	Design separates particle and dissolved effects	[72]
Lignin/DMA–carrageenan–PVP hydrogel	Phytotoxicity	*Zea mays* germination index	Rated non-phytotoxic	[67]
Nyssa gum-*g*-poly(AA) hydrogel	Phytotoxicity of treated water	Seed germination	100% germination, as in distilled water	[68]
Alginate–*O. ficus-indica* mucilage hydrogel	Phytotoxicity	Lettuce seeds	No toxicity observed	[69]

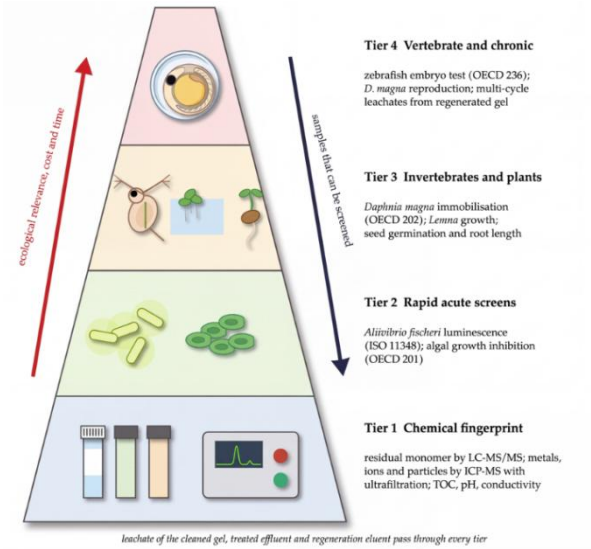


Figure 04: Proposed tiered scheme for testing hydrogel adsorbents and the water they treat, from leachate chemistry to vertebrate and chronic assays. Test guidelines: ISO 11348 [78], OECD 201 [79], OECD 202 [80] and OECD 236 [81].

6. SAFE-BY-DESIGN AND MITIGATION

Most of the problems above can be reduced at the design stage. Figure 5 groups the options into four levers.

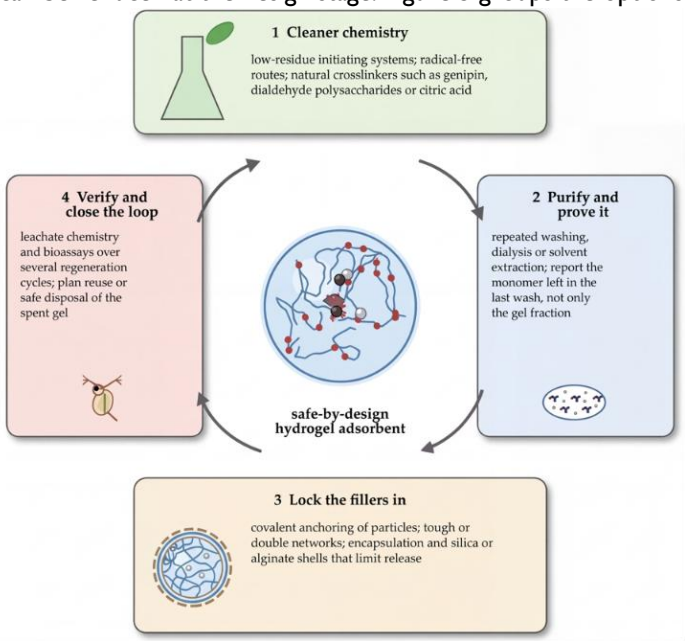


Figure 05: Four safe-by-design levers for hydrogel adsorbents: cleaner chemistry, verified purification, firm anchoring of fillers, and verification with planned end of life.

6.1. Cleaner chemistry

The simplest lever is the polymerization itself. Changing the initiating system lowers residual monomer without new equipment [19,27]. Gallium nanoparticles and MoS_2 nanosheets have served as initiators for polyacrylamide gels with low monomer content [20]. Radical-free routes go further. Carbodiimide-mediated crosslinking of preformed polymers avoids acrylamide and bisacrylamide altogether and leaves only non-toxic by-products [82]. For polysaccharide gels, natural crosslinkers such as citric acid, tannic acid and vanillin are realistic options [4], as are dialdehyde polysaccharides [18,65] and even gold nanoparticles [16]. Each comes with trade-offs in strength and cost, and each still needs testing.

6.2. Purification that is measured, not assumed

Washing is the norm, but hardly checked. In automated studies, dialysis cleared up to 99% of unreacted monomer [54]. Ethanol precipitation cut down gelatin methacryloyl to less than 30 ppm of residues in two days instead of seven [83]. Ethanol extraction and membrane dialysis also proved successful for lignin–acrylamide copolymers [57], and an extruded hybrid gel had <200 ppm of residual monomer after purification [84]. The slow tail as described by Brynda et al. [40] calls for a final check: analyse the last wash water, not only the gel. The monomer is also carried in the wash water which is a waste stream that should not go straight to the drain.

6.3. Locking the fillers in

Fillers need to be anchored, not only trapped. Tough, self-healing networks keep the nanomaterials in place through repeated use [5,85]. Silica impregnation increased the durability of magnetite gel beads without affecting their capacity [86] and alginate immobilisation reduced metal leaching from a Cu – Fe catalyst [47]. Encapsulation can aid although, as mentioned above, it may just delay release [42]. Where some loss is unavoidable, a polishing step after the adsorber is a sensible protection [39].

6.4. Verification and end of life

The final lever is to inspect, and to arrange the exit. Regeneration eluents must be compatible with the gel chemistry; strong base is not compatible with amide crosslinks [15,45]. Used gels need a declared method, whether reuse, recovery or safe disposal [10,49,87]. Given what is known about superabsorbent residues in soil, the case for agricultural reuse of dye-loaded or acrylate gels should be made, not assumed [8,48].

7. RESEARCH GAPS AND A MINIMUM REPORTING SET

This survey reveals some gaps. Firstly, there are hardly any papers on hydrogel adsorbents that report residual monomer in a leachate of the completed, washed material. Second, the release of nanofillers is rarely monitored throughout regeneration cycles or in real water. Third, knowledge of crosslinker toxicity is mostly from mammalian cells, with almost none from aquatic organisms. Fourth, safety claims depend upon seed germination. Fifth, we ignore mixes. The treated effluent contains the residual pollutant, the leachates and any transformation products all together and it is their combined effect that organisms encounter. Regulation is part of the picture too. Polymers are generally treated as being of low environmental concern and have been exempt from registration under REACH, an exemption that the European Commission has been reviewing [13]. Adsorbent gels fall in the same blind area. They are neither a chemical dosed to water, like a flocculant, nor a regulated product. Life-cycle metrics for spent adsorbents are also still being defined [49], so the burden of the washing water, the eluents and the spent gel seldom enters any comparison of materials. None of thisneeds exotic tools. Table 4 proposes a minimum set of items that would let readers judge whether a gel adds to the water it cleans. Most items cost little compared with the synthesis work already done.

Table 04: Proposed minimum reporting set for leaching and ecotoxicity of hydrogel adsorbents.

Item	Minimum practice	Reason
Residual monomer and crosslinker	Quantify in the last wash water and in a 24–72 h leachate of the washed gel by LC-MS/MS or a validated HPLC method	The gel fraction shows what stays, not what moves
Leaching conditions	State solid-to-liquid ratio, time, temperature, pH, hardness and ionic strength	Release depends strongly on the water matrix
Nanofiller release	Total and dissolved metal by ICP-MS after 0.22 µm filtration or ultrafiltration, at every cycle	Dissolved ions drive most of the toxicity
Regeneration	Analyse the spent eluent and the first effluent after regeneration	Acid and base pulses release fillers and break links
Bioassays	At least two trophic levels (bacteria or algae plus *D. magna*), with clean-water and adsorbent-only controls	Seed germination alone is too coarse
Dose–response	Test undiluted effluent and a dilution series; report EC_{50}, LOEC or toxic units	Allows comparison and risk ranking
Ageing and fragments	Mass loss and particle counts after the last cycle	Gel debris may persist like microplastics
End of life	State the reuse or disposal route and test leaching of the spent gel	Avoids moving the problem to soil or landfill

One more point is worth making. Journals and reviewers could ask for these data as routinely as they ask for isotherm fits. Until they do, the word eco-friendly in the title of an adsorbent paper tells the reader very little.

8. CONCLUSION

Hydrogel adsorbents clean water well, but they are not inert. We evaluate the findings in this paper and show three kinds of species that can migrate from the gel into the treated water: residual monomers like acrylamide, unreacted crosslinkers and initiator fragments, and nanofillers together with their dissolved ions. Release is not a one-time thing. It peaks after synthesis, comes back with each acid or base regeneration and continues while the wasted gel matures. The toxicity results were a mixed blessing. Silver ions are active at <1 μg L−1 therefore filler leaching is directly relevant to aquatic life. Acrylamide, however, is acutely hazardous only at mg L−1. Bioassays alone will therefore miss acrylamide at the drinking-water limitations and chemical analysis is needed. Present safety claims, largely based on seed germination, cannot address either issue. An economical tiered method correlating leachate chemistry with testing on at least two trophic levels should be routine practice. Combined with cleaner synthesis, validated purification and solid anchoring of fillers, it would make hydrogel adsorbents fulfilll their promise without any hidden footprint.

9. CONFLICT OF INTEREST

The authors declare no conflicts of interest.

10. ETHICAL APPROVAL AND INFORM COPNSENT

Not applicable.

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