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REVIEW ARTICLE

Integrated Extraction–Adsorption of Anthocyanins from Aronia melanocarpa: Experimental Evidence, Dynamic Mass Transfer, and Process–Wide Recovery Assessment

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ABSTRACT: Black chokeberry *(Aronia melanocarpa)* is rich in cyanidin glycosides, yet improved extraction yield does not necessarily lead to greater recovery in the final product. This review critically evaluates evidence from integrated extraction–adsorption processes, stand-alone resin purification, and fixed-bed mass-transfer studies. Particular attention is given to extract composition, three-phase dynamic mass balances, transport resistance at multiple scales, and competitive adsorption. Published work has reported increases of more than 25% in phenolic extraction yield and more than twofold in process productivity relative to sequential extraction–adsorption. In a study of chokeberry skin waste, anthocyanin yield increased from 5.25 to 6.34 mg/g dry pomace, while the anthocyanin mass fraction of the dried extract rose from 19.9% to 28.4%. A separate resin purification study recovered approximately 96% of the anthocyanins present in the feed extract; this figure represents step recovery rather than overall recovery from the raw material. Although adsorption demonstrably captures anthocyanins already released into solution, evidence remains insufficient to establish whether lowering their liquid-phase concentration also promotes further release from plant tissue. Changes in circulation, mixing, and chemical loss have not been quantitatively disentangled. Hydrodynamically matched controls, component-resolved mass balances across individual phases, and independent validation of coupled transport models are therefore needed. Product integrity, resin regeneration, and resource consumption should be considered alongside recovery when assessing industrial relevance.

Keywords: Aronia Melanocarpa; Cyanidin Glycosides; Integrated Extraction–Adsorption; Dynamic Mass Balance; Macroporous Resin; Mass-Transfer Resistance; Overall Process Recovery

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1. INTRODUCTION

Black chokeberry *(Aronia melanocarpa (Michx.) Elliott)* is a source of anthocyanins, proanthocyanidins, phenolic acids, and flavonols, making it a promising feedstock for natural colorants and phenolic ingredients [1, 2]. Its anthocyanins are dominated by cyanidin-3-O-galactoside and cyanidin-3-O-arabinoside, with smaller amounts of the corresponding glucoside and xyloside [1]. Differences in glycosylation may affect solvation, partitioning onto resins, and stability during processing. In aqueous media, anthocyanins alsoundergo pH-dependent, reversible structural transformations and may undergo irreversible degradation upon heating or oxidation [3, 4]. An effective recovery process must therefore promote release from the plant matrix while limiting losses during subsequent separation and processing.

Ultrasound-assisted extraction, enzyme treatment, pressurized liquid extraction, and deep eutectic solvents have all been investigated as means of improving anthocyanin extraction [5–9]. These approaches can modify tissue structure, intensify fluid-phase transport, or alter the solvent environment. However, a higher extraction yield does not

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necessarily produce a higher final product recovery. Crude chokeberry extracts contain co-extracted phenolics and soluble matrix constituents; their composition, viscosity, acidity, and residual extraction solvents may affect resin adsorption, elution, and the stability of individual compounds. Extraction conditions therefore need to be evaluated in relation to subsequent purification.

Integrated extraction–adsorption connects release from plant tissue with simultaneous capture from the liquid phase and may reduce the time or processing burden associated with fully separated operations. Galván D'Alessandro et al. [10] investigated simultaneous extraction and adsorption of chokeberry phenolics, while Vauchel et al. [11] examined scale-up of the process. Using chokeberry skin waste, Wathon et al. [12] reported an increase in anthocyanin yield from 5.25 to 6.34 mg/g dry pomace and an increase in the anthocyanin mass fraction of the dried extract from 19.9% to 28.4%. These findings demonstrate that process configuration can affect both recovery and enrichment. They do not, however, independently quantify the contributions of enhanced release, adsorption capture, or changes in degradation.

Removal of anthocyanins from the liquid phase could, in principle, sustain the concentration gradient driving transfer from plant tissue when release depends on the external concentration. Integrated operation also changes circulation, mixing, and residence time, while resin loading, competition among components, and chemical degradation may further influence the outcome. Zhu et al. [13] described transport and intraparticle diffusion during fixed-bed adsorption and desorption of anthocyanins. Their findings inform the behavior of a stand-alone adsorption unit but do not establish that concentration-mediated enhancement of extraction occurs in a coupled process.

This review examines evidence for integrated extraction–adsorption of chokeberry anthocyanins, distinguishing direct integration experiments from stand-alone resin purification and fixed-bed transport studies. It then considers how upstream extraction conditions affect resin capture and uses a three-phase dynamic mass balance to examine the relationships among release, adsorption, and chemical loss. Finally, it outlines a validation and evaluation framework centered on component-resolved material distributions, breakthrough behavior, and final-product recovery. The discussion synthesizes published studies and does not present original experimental measurements.

2. EXPERIMENTAL EVIDENCE AND LIMITS OF MECHANISTIC INTERPRETATION

2.1. Process Configurations and Operating Principles

Conventional anthocyanin production typically begins with solvent extraction from plant tissue, followed by solid–liquid separation, resin adsorption, and elution. This configuration allows the operating conditions of each step to be optimized separately. In some extraction systems, however, accumulation of solutes in the liquid phase may reduce the net driving force for further release. Integrated extraction–adsorption seeks to remove target compounds from solution while they are being released, thereby coupling the two operations in time.

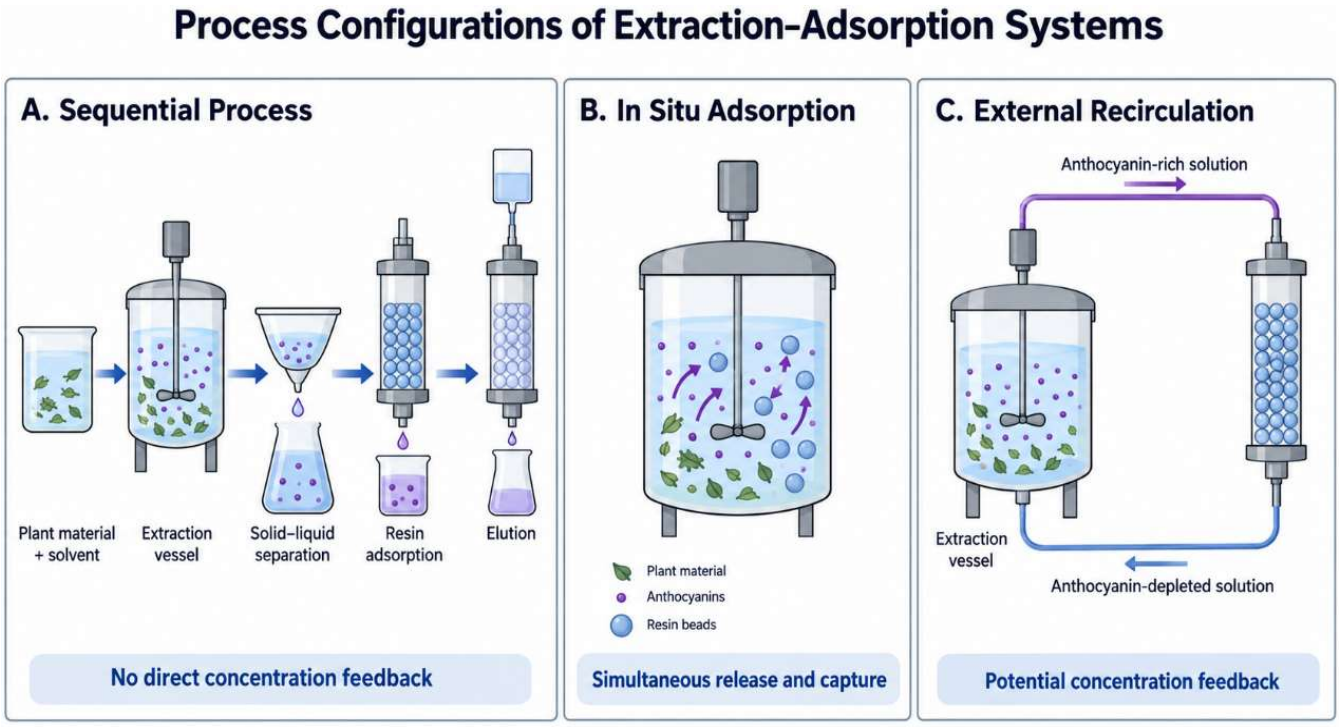


Fig. 1. Sequential, in situ, and externally recirculated extraction–adsorption configurations.

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Integrated processes can be classified by spatial configuration as in situ contact systems or external recirculation systems (Figure 1). In the former, plant particles and the adsorbent occupy the same vessel; in the latter, extract flows through a separate adsorption bed before returning to the extraction vessel. Both arrangements can change the liquid-phase concentration, but they differ in solids separation, resin loading, and fluid-transport requirements. External recirculation may itself improve mixing and solid–liquid contact. Without a control operated under comparable recirculation conditions, the entire observed improvement cannot be assigned to adsorptive removal.

2.2. Effects on Target Recovery and Product Composition

Galván D'Alessandro et al. [10] coupled the extraction of chokeberry phenolics with XAD7HP resin adsorption and reported a phenolic extraction yield more than 25% higher than that obtained by sequential operation; process productivity more than doubled. These are direct comparisons of process performance, but phenolic extraction yield should not be equated with the recovery of individual anthocyanins. Productivity also depends on processing time and the definition of included operations. Consequently, these data cannot isolate the effect of reduced liquid-phase concentration on release from the tissue.

Wathon et al. [12] compared conventional batch extraction of chokeberry skin waste with a recirculated extraction–adsorption process. Anthocyanin yield increased from 5.25 to 6.34 mg/g dry pomace (approximately 20%), while the anthocyanin mass fraction of the dried extract rose from 19.9% to 28.4%, an increase of 8.5 percentage points (approximately 43% relative). Product composition also differed: the reported relative proportion of cyanidin aglycone changed from 50.7% to 32.0%, whereas cyanidin-3-O-galactoside changed from 38.8% to 45.7%. The original authors discussed differences in acid-catalyzed breakdown as a possible explanation. Relative proportions alone, however, cannot demonstrate reduced absolute loss of any particular compound, because adsorption selectivity, desorption, and recovery may also alter composition.

Together, these studies show that integration can improve product output and modify composition under specific feedstock and operating conditions. Their feedstocks, analytes, and denominators differ, however, and the reported changes should not be interpreted as interchangeable measures of process improvement. Evidence from direct integration must also be distinguished from stand-alone purification of previously extracted material and independent fixed-bed kinetics (Table 1).

2.3. Complementary Evidence from Scale-up and Stand-alone Adsorption

Vauchel et al. [11] scaled up an integrated extraction–adsorption process for antioxidant phenolics from chokeberry waste by a factor of 50 in terms of feedstock, adsorbent, and solvent use: feedstock and adsorbent quantities increased from 4 to 200 g, and solvent volume from 0.8 to 40 L. Performance at the larger scale was reported to be comparable to that at laboratory scale. Within the conditions examined, processing time had a marked influence, whereas recirculation flow rate did not have a statistically significant effect. Parameter sensitivity thus depends on equipment and the operating range; a nonsignificant flow-rate effect does not establish the absence of diffusion resistance. The study also does not replace long-term testing of continuous operation and repeated resin regeneration.

Roda-Serrat et al. [14] treated a previously prepared chokeberry pomace extract using XAD7HP resin and recovered approximately 96% of the anthocyanins initially present in the purification feed after adsorption and desorption. The denominator is the anthocyanin mass entering purification, making this a step-recovery value. The result does not describe anthocyanins remaining unextracted in the raw material and cannot be used as the overall raw-material-to-product recovery.

Zhu et al. [13] investigated fixed-bed adsorption and desorption of chokeberry anthocyanins using a model incorporating axial convection–dispersion and intraparticle diffusion, including comparisons with and without ultrasound. This work provides a framework for analyzing bed-scale transport. Nevertheless, the fixed-bed feed investigated was not equivalent to a recirculating extract continuously supplied with compounds from plant tissue. Applying that model to an integrated system would require time-resolved measurements of feed composition, bed effluent, and resin loading.

2.4. Current Understanding and Unresolved Mechanistic Questions

Direct integration studies compare recovery, enrichment, and productivity under defined operating conditions. Stand-alone resin separation and fixed-bed studies provide complementary information on adsorption–desorption behavior and bed transport. Quantitative connections among release from raw material, liquid-phase residence, resin capture, eluted product, and chemical losses nevertheless remain inadequate.

This gap is closely associated with the unsteady nature of integrated operation. Target compounds are released continuously, resin loading increases, and concentrations of anthocyanins and competing solutes evolve over time. A low liquid-phase concentration at a given time does not reveal whether extraction is faster, adsorption is faster, or degradation is greater. Identifying the source of any integration benefit requires contemporaneous measurements of the material remaining in the plant residue and distributed among the liquid, resin, and final product under comparable conditions.

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Table 1. Process configurations, operating conditions, and principal findings of representative studies.

Study	Configuration	Feedstock and scale	Resin / medium	Key operating features	Main findings (original reporting basis)
Galván D'Alessandro et al. [10]	Direct integration (simultaneous extraction and adsorption)	Chokeberry feedstock; laboratory scale	XAD7HP	Integrated versus sequential extraction–adsorption	Phenolic extraction yield >25% higher; process productivity >2-fold
Wathon et al. [12]	Recirculated extraction–adsorption versus conventional batch extraction	Skin waste; dry pomace basis	Not specified in manuscript	Integrated recirculation	Anthocyanins: 5.25 → 6.34 mg/g dry pomace; anthocyanin mass fraction in dry extract: 19.9% → 28.4%
Vauchel et al. [11]	Scale-up with external recirculation	4 → 200 g feedstock; 0.8 → 40 L solvent	Verify against original article	50-fold scale-up; processing time influential	Performance comparable to laboratory scale
Roda-Serrat et al. [14]	Sequential purification (stand-alone adsorption–desorption)	Pomace extract	Verify against original article	Defined adsorption–desorption conditions	Approximately 96% step recovery of feed-extract anthocyanins
Zhu et al. [13]	Fixed-bed transport modeling	Chokeberry anthocyanin fixed-bed feed	Macroporous resin; type to verify	Axial convection–dispersion and intraparticle diffusion	Mechanistic description of adsorption–desorption

3. INTERACTIONS AND TRADE-OFFS BETWEEN EXTRACTION INTENSIFICATION AND RESIN SEPARATION

3.1. Effects of Intensified Extraction on Release and Crude-Extract Composition

Release of chokeberry anthocyanins entails penetration of the solvent into plant tissue, dissolution of the compounds, and transport within the tissue and across the solid–liquid interface. In aqueous ethanol, solvent composition, acidity, and temperature affect anthocyanin solubility but also the co-extraction of other phenolics and soluble matrix materials. Extraction optimization should therefore consider not only the amount recovered in the extract but also its suitability as a resin feed.

Enzyme-assisted, ultrasound-assisted, and pressurized liquid extraction act through partly distinct mechanisms. Kim et al. [8] treated chokeberry pomace with a combination of pectin-related enzymes and cellulase and reported approximately 50.7% more total anthocyanins than with the solvent-extraction control. The response may reflect cell-wall modification, although enzymatic hydrolysis products may also affect clarification. Ultrasound can improve tissue–solvent contact through cavitation and fluid disturbance; Galván D'Alessandro et al. [5] observed enhanced release kinetics during the early stage of extraction. Vázquez-Espinosa et al. [15] reported different optimal conditions for total phenolics and total anthocyanins, indicating that process intensification may alter the release of several compound classes simultaneously.

Pressurized liquid extraction controls temperature, pressure, and solvent flow to promote contact between the extraction medium and plant tissue. Combining ultrasound with pressurized liquid extraction, Andrade et al. [6] reported an anthocyanin extraction proportion of 88.0% after 45 min at 70 °C, 180 bar, and an optimized citric acid concentration of 1.5% (w/w), among other conditions. The 88.0% value refers to extraction relative to the reference basis used in that study, not product purity or overall process recovery. Relative to batch extraction, flowing solvent also changes the

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concentration and flow rate of the outlet stream, and therefore the loading imposed on a downstream resin bed. Its suitability for coupled purification has yet to be tested independently.

Deep eutectic solvents primarily influence anthocyanin solubility and stability by altering the solvation environment. Lv et al. [9] compared ultrasound-assisted extraction using ethanol, aqueous biphasic systems, and deep eutectic solvents; their optimized deep eutectic solvent system yielded approximately 16.6 mg/g total anthocyanins, exceeding the two comparators within that study. The figure is specific to its sample and analytical basis and should not be ranked directly against mg/g values from unrelated studies. Solvent viscosity, residual constituents, and solute–solvent interactions may affect adsorption and intraparticle transport; these effects require measurement of partitioning and breakthrough with the actual extract.

The common consequence of extraction intensification is a change in the stream entering purification, not merely an increase in one target analyte. Table 2 separates reported extraction outcomes from properties that might affect downstream separation. Entries concerning bed fouling, loading strategy, and net recovery represent questions for further testing rather than demonstrated effects in an integrated chokeberry system.

3.2. Solvent Environment and Matrix Constituents in Resin Adsorption

During resin loading, anthocyanins must partition from the extract into the solid adsorbent. Their capture by nonionic macroporous resins depends on pore architecture, surface chemistry, solvent composition, and other solutes. Hydrophobic and van der Waals interactions, hydrogen bonding, and interactions between aromatic moieties may contribute [17, 18]. None can be established from a single adsorption-kinetics fit, and a static capacity measured in water need not represent dynamic performance in a complex extract.

Changing the ethanol fraction can alter both anthocyanin solvation and its partitioning onto a resin. Deep eutectic solvents may also affect adsorption and pore transport through their solvation behavior and viscosity [9, 16, 17]. Whether dilution or solvent exchange is justified should be assessed from net target recovery, additional material losses, and solvent-processing requirements. The available evidence does not establish that deep eutectic solvents universally enhance or suppress resin capture.

Phenolic acids, flavonols, and proanthocyanidins in crude chokeberry extracts may compete with anthocyanins for accessible adsorption regions [1, 2, 19]. Sugars, organic acids, and pectin may instead act mainly through changes in pH, viscosity, colloidal properties, or bed pressure drop. Matrix effects should not all be attributed to competition for adsorption sites: molecular selectivity and changes in fluid flow or transport need to be considered separately.

The principal cyanidin glycosides share the same aglycone scaffold but differ in sugar moieties, which may influence solvation and resin partitioning. Resin screening should therefore extend beyond static total-anthocyanin capacity to measurements of individual glycoside breakthrough and desorption profiles before claims of selectivity are made.

Table 2. Extraction strategies, their effects on crude-extract properties, and questions concerning downstream resin compatibility.

Extraction strategy	Reported extraction-side outcome	Feed properties potentially affected	Unresolved issue for resin purification
Enzyme-assisted [8]	Approximately 50.7% higher total anthocyanin extraction than solvent control	Pectin hydrolysates, colloids, and nontarget compounds	Clarification, pressure drop, and selectivity
Ultrasound-assisted [5, 15]	Enhanced early release; different optimal conditions for phenolics and anthocyanins	Fine particles, co-extracted phenolics, thermal history	Loading requirements and bed fouling
Ultrasound + pressurized liquid [6]	88.0% extraction in 45 min at 70 °C, 180 bar, and 1.5% (w/w) citric acid	Temperature, flow, acidity, and outlet concentration	Cooling, pressure change, and bed loading
Deep eutectic solvents [9, 16, 17]	Approximately 16.6 mg/g in an optimized system (basis defined in original study)	Solvation, viscosity, and residual solvent components	Net recovery after direct loading, dilution, or solvent exchange

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3.3. Transport Trade-offs during Dynamic Adsorption and Desorption

Resin adsorption involves transfer from the bulk liquid to particle surfaces, diffusion within the pores, and local adsorption equilibria. The relative resistance of these steps depends on particle size, flow rate, solvent viscosity, and resin loading. High equilibrium capacity does not guarantee rapid capture; even a good fit of a pseudo-second-order kinetic expression or a single breakthrough curve cannot, by itself, establish chemisorption or identify a unique rate-limiting step [20, 21].

Increasing flow rate through a fixed bed may improve external-film mass transfer while reducing the empty-bed contact time, potentially causing earlier breakthrough. The net response depends on resin capacity, particle size, flow distribution, and feed composition. It should be evaluated using breakthrough profiles, dynamic working capacity, and pressure drop rather than inferred from static saturation capacity.

Zhu et al. [13] compared adsorption and desorption dynamics in a fixed bed under ultrasound treatment. The operational objective differs between stages: adsorption seeks to retain solutes effectively, while desorption aims to transfer retained compounds into the eluent. A given intensification measure therefore cannot be judged by the same criterion in both stages. In particular, earlier breakthrough should not automatically be interpreted as a decrease in equilibrium resin capacity, nor faster desorption as an increase in overall process recovery.

Eluent composition and volume affect anthocyanin desorption, the concentration of the eluate, co-elution of other phenolics, and the need for downstream concentration. The approximately 96% value reported by Roda-Serrat et al. [14] is an adsorption–desorption step recovery under the specified conditions; it is distinct from final-product purity and solvent efficiency.

3.4. From Intensified Unit Operations to Process Integration

Extraction transfers target compounds out of the plant matrix; adsorption transfers them to the resin; and desorption transfers them into the eluent. These operations differ in their preferred solvent environments and transport rates. Conditions that optimize one stage may therefore be suboptimal for the overall process.

In directly integrated systems, release from the tissue and progressive resin loading occur simultaneously, while both anthocyanin concentration and the composition of competing solutes change over time. Extraction and adsorption should consequently be assessed within a shared material-balance framework rather than through extraction yield and static adsorption capacity considered in isolation.

4. DYNAMIC MASS TRANSFER AND MECHANISTIC ANALYSIS OF COUPLED EXTRACTION–ADSORPTION

4.1. Dynamic Mass Balances and the Release–Capture Relationship

In a sequential process, target compounds are generally released into the extract before contacting the adsorbent. In an integrated process, release and capture can occur together and are connected through the amount of solute present in the liquid phase. It is essential to distinguish capture of material already released from additional release caused by that capture.

Resin adsorption initially redistributes released anthocyanins between phases. Lowering the liquid concentration can accelerate subsequent net release only if release is sensitive to the external concentration. A higher resin loading is therefore not direct evidence of concentration-driven extraction enhancement. Testing for such feedback requires, at minimum, a hydrodynamically matched control without adsorption and measurements of intact anthocyanins remaining in the plant residue.

For a conceptual analysis, the system may be divided into three control volumes: plant tissue, liquid, and resin (Figure 2). Let Ms、M_l和Mr denote the masses of intact anthocyanins in the tissue, liquid, and resin phases, respectively; r_e denote the rate of release from tissue to liquid; r_a denote the net rate of adsorption onto resin; and $r_{d,l}$ and $r_{d,r}$ denote the irreversible degradation rates in the liquid and resin phases, respectively.

The quantities Ms, M_l , and M_r are masses (e.g., mg);r_e,r_a,$r_{d,l}$, and$r_{d,r}$ are rates of mass transfer or loss (e.g., mg/min). All quantities should be expressed on a common analytical basis for intact anthocyanins. When individual anthocyanins are tracked, the mass balances should be written separately for each compound before the results are summed.

For an idealized closed system with constant liquid volume, lumped phase inventories, no external material input or output, and negligible degradation within the plant tissue, the following balances apply to intact anthocyanins. The net adsorption rate may be negative if desorption occurs. Equations (1) to (4) are statements of mass conservation, not empirically validated kinetic expressions for release or adsorption. Plant-tissue phase:

$$\frac{dM_s}{dt} = -r_e \quad (1)$$

Liquid phase:

$$\frac{dM_l}{dt} = r_e - r_a - r_{d,l} \quad (2)$$

Resin phase:

$$\frac{dM_r}{dt} = r_a - r_{d,r} \quad (3)$$

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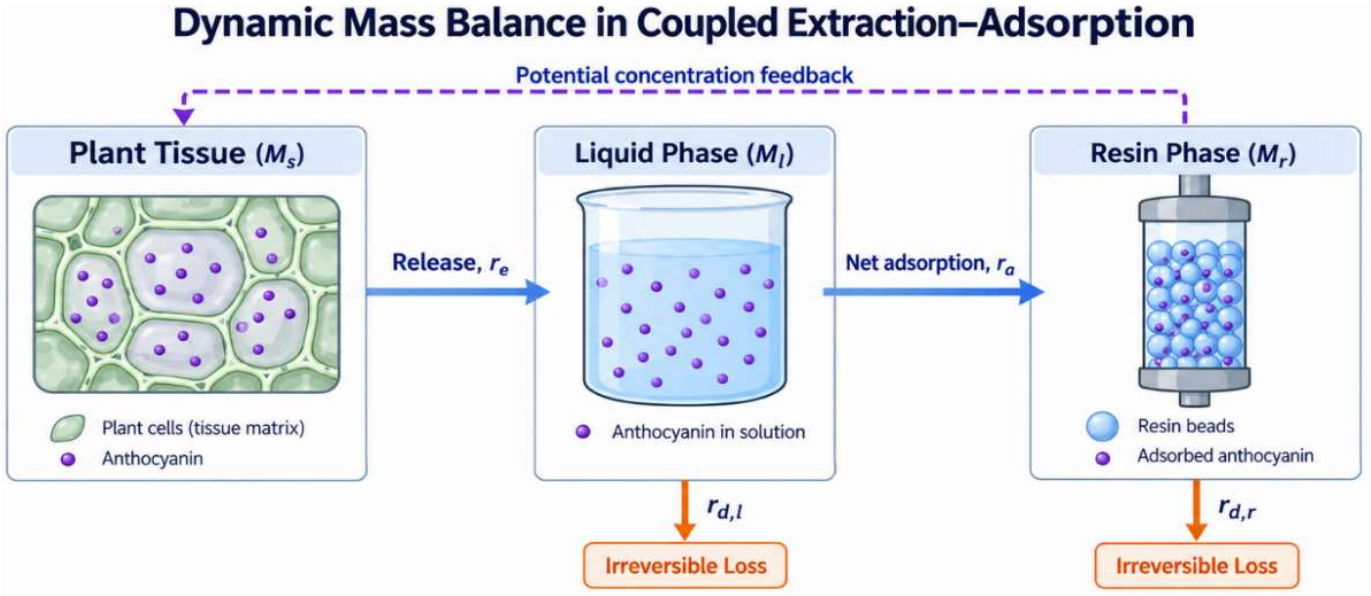


Fig. 2. Control volumes and mass flows in a coupled extraction–adsorption system.

Summing Equation (1) to (3) gives the overall balance for intact anthocyanins in the coupled system:

$$\frac{d(M_s+M_l+M_r)}{dt} = -(r_{d,l} + r_{d,r}) \quad (4)$$

Equation (4) shows that, under the closed-system assumptions, release and adsorption redistribute intact anthocyanins among the three control volumes, while irreversible degradation in the liquid and resin phases accounts for their total mass loss. The equation does not establish that integration reduces degradation. It also excludes degradation within plant tissue, sampling, equipment holdup, and continuous inlet or outlet streams; these contributions must be added when analyzing an actual device.

In an external recirculation apparatus, liquid held within the packed bed and piping may be non-negligible. Appropriate control volumes must include inlet, outlet, and sampling mass flows. Net retention in a bed should be evaluated from its inlet and outlet concentrations and measured flow rate, with changes in bed liquid inventory accounted for. Equation (4) cannot be applied without modification to close a material balance for an open or intermittently sampled system.

A decline in liquid-phase anthocyanin concentration may reflect faster resin capture, slower tissue release, or greater chemical loss. A single concentration–time profile for the circulating liquid cannot discriminate among these explanations. Even if the liquid concentration remains low, additional release of intact compounds must be demonstrated by directly measuring the residual plant material.

Concentration feedback can increase release only if changes in external concentration measurably affect the net transfer from plant tissue. If intratissue diffusion or binding to the matrix dominates the resistance, further lowering the liquid concentration may yield little benefit. Increasing resin loading, competition from co-extracted compounds, and transport limitations may also prevent capture from keeping pace with release.

A practical recirculating fixed-bed process additionally contains liquid holdup within the bed and piping, sampling losses, and switches between adsorption and elution. Measurements must therefore include the plant residue, circulating liquid, resin inventory, bed effluent, eluted product, and other identifiable losses, without counting material repeatedly as it circulates through a given location. Only a component-resolved, reasonably closed balance can distinguish increased release from improved capture or reduced chemical loss.

4.2. Multiscale Mass-Transfer Resistance and Its Evolution

Coupled extraction–adsorption involves transport at the scales of plant tissue, circulating liquid, and resin particles (Figure 3). Their pathways and characteristic response times differ, and their relative importance may change with feedstock structure, flow rate, resin loading, and extract composition. Intensifying a local step improves the process only when that step constrains delivery of the target compounds to the final product.

At the plant-tissue scale, release depends on cell-wall structure, internal diffusion pathways, and transport across the solid–liquid interface. Comminution, enzymatic treatment, and ultrasound may shorten effective diffusion paths or improve interfacial contact. Galván D'Alessandro et al. [5] observed enhancement of early-stage extraction kinetics by ultrasound. Nevertheless, apparent diffusivities

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fitted to bulk release curves often incorporate several transport contributions and should not be interpreted as intrinsic diffusion coefficients within a specific cellular structure.

External recirculation affects both mixing and liquid renewal in the extraction vessel and contact time and throughput in the fixed bed. Increasing flow rate may improve extraction-side transport while advancing adsorption-side breakthrough. This trade-off requires comparison of inlet and outlet concentration profiles at the same resin loading and bed configuration.

At the resin-particle scale, anthocyanins are transported across the external liquid film, through pores, and to adsorption regions. When intraparticle transport is relatively slow, stronger fluid agitation may not improve dynamic working capacity. Changes in resin loading, competing solutes, and flow distribution can also influence breakthrough curves [20, 21].

The dominant limitation may change during operation. Early tissue release, increasing resin loading as the process progresses, and transport of residual components at later stages could each become important. These are possible scenarios to be tested, not an experimentally established sequence of rate-controlling steps.

The purpose of multiscale modeling is thus not simply to list possible resistances, but to use experiments with different parameter sensitivities to determine when and under which operating conditions a particular intensification strategy improves overall recovery.

4.3. Multicomponent Competitive Adsorption and Dynamic Selectivity

Real chokeberry extracts contain phenolic acids, flavonols, proanthocyanidins, and other matrix constituents alongside anthocyanins [1, 2]. Because these compounds may differ in their release rates and resin affinities, the composition of the circulating feed changes over time. Apparent selectivity in a resin bed depends not only on the adsorbent but also on time-dependent feed composition and competition.

Competition may occur among adsorbable phenolics. If a more strongly retained component displaces one previously adsorbed with lower affinity, the effluent concentration of the latter may increase relative to its incoming concentration. Such displacement requires component-specific breakthrough evidence that accounts for changing feed composition, analytical uncertainty, and degradation. Variation in total anthocyanins or total phenolics alone cannot establish this mechanism.

Differences in the sugar moieties of the four principal cyanidin glycosides may affect solvation and resin partitioning. Whether these differences produce useful separation selectivity depends on resin architecture and the liquid environment. The compositional changes observed by Wathon et al. [12] could reflect both chemical transformation and selective recovery; they do not identify the dominant step for any one glycoside.

The effects of sugars, organic acids, and pectin on bed performance may arise primarily from pH, viscosity, colloidal behavior, and pressure drop rather than direct competition for anthocyanin adsorption regions. Chemical competition should therefore be distinguished from engineering effects on flow and transport.

Mechanistic tests could begin with single-solute and binary systems and progress to actual crude extracts. Dynamic breakthrough, resin loading, and the masses eluted should be measured for individual cyanidin glycosides and representative co-extracted phenolics. Reporting absolute masses rather than only composition percentages is essential for distinguishing selective capture, competitive displacement, and chemical losses.

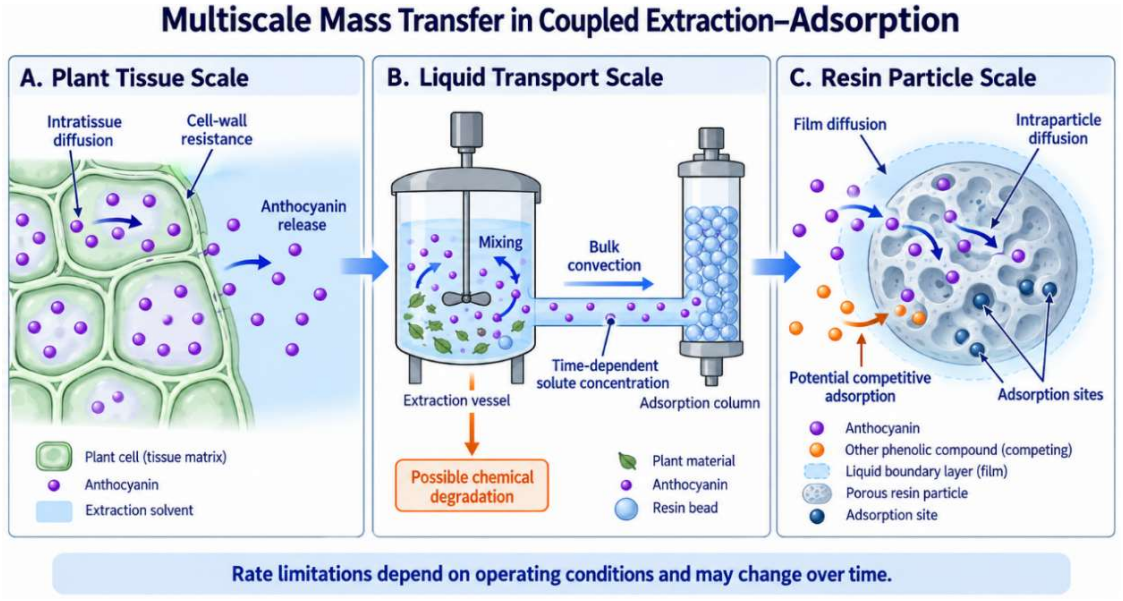


Fig. 3. Multiscale transport pathways and potential rate limitations in the coupled process.

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4.4. Coupled-Process Modeling and Mechanistic Validation

Thomas, Bohart–Adams, and Yoon–Nelson equations are commonly used to correlate fixed-bed breakthrough curves, but their parameters depend on model assumptions and operating conditions and should not automatically be treated as independent physical constants [20, 21]. Mechanistic models that explicitly represent axial convection–dispersion, film mass transfer, and intraparticle diffusion can describe transport pathways more directly. Zhu et al. [13] provide an example for an independent fixed bed, but their model cannot directly predict a recirculating system continuously supplied by release from plant tissue.

An external-recirculation model should describe tissue release and bed adsorption separately, then couple them through circulation flow and the inlet and outlet mass flows of each unit. A spatially distributed model should account for bed liquid holdup, axial dispersion, and intraparticle diffusion; a lumped model should justify neglecting those effects. Model complexity should be constrained by independent observables and parameter identifiability.

Several processes can produce similar circulating-liquid concentration profiles: adsorption may accelerate, release may diminish, or degradation may intensify. Simultaneously fitting multiple diffusion, adsorption, and degradation parameters to one profile may yield a high goodness of fit without unique parameter identification. Credible interpretation requires independent parameter measurements, mass-balance closure, and predictive tests under operating conditions excluded from fitting.

One practical strategy is to constrain key parameters using separate extraction kinetics, static adsorption, and fixed-bed breakthrough experiments, then compare hydrodynamically matched recirculation controls without adsorbent against the actual coupled process. Systematic changes in feedstock particle size, resin particle size, flow rate, bed height, and inlet composition can help test sensitivity to different transport steps and prediction across operating conditions. The observables and interpretative limits of the relevant model classes are summarized in Table 3. The objective of modeling should be to identify processes that constrain recovery, not merely to reproduce previously measured curves. Phase-resolved mass balances, independently constrained parameters, and validation across operating conditions form a minimum evidential framework for mechanistic interpretation of coupled extraction–adsorption.

5. WHOLE-PROCESS QUALITY ASSESSMENT AND ENGINEERING APPLICABILITY

5.1. Recovery Metrics and Evaluation Boundaries

The objective of extraction–adsorption is to obtain intact anthocyanins in a product that meets its intended specifications. Extraction yield, equilibrium capacity, dynamic working capacity, step recovery, and product purity refer to different process stages and denominators and must be defined before comparisons are made. A favorable result for one metric is not sufficient evidence of superior overall recovery.

Table 3. Key observables, interpretative capabilities, and limitations of models at different process scales.

Process level	Representative model / parameter	Key measurable quantities	Not established by these data alone
Release from tissue	Empirical release rate; apparent effective diffusivity	Residual compound masses, release curves, particle size	Intrinsic diffusivity or a unique cell-disruption mechanism
Static adsorption	Langmuir/Freundlich isotherms; kinetic correlations	No-resin controls, particle size, agitation, individual solute concentrations	A good pseudo-second-order fit does not prove chemisorption control
Fixed-bed capture	Empirical breakthrough models; convection–dispersion and pore diffusion	Time-dependent inlet/outlet profiles, bed height, pressure drop	A unique resistance identified from a single breakthrough curve
Multicomponent competition	Competitive isotherms and dynamic component balances	Component-specific breakthrough and elution of glycosides and phenolic acids	Displacement inferred solely from total phenolic concentration
Whole-process coupling	Phase-resolved mass balances linked by flow	Residue, circulating liquid, resin, eluate, and losses	Low liquid-phase concentration does not imply high final recovery

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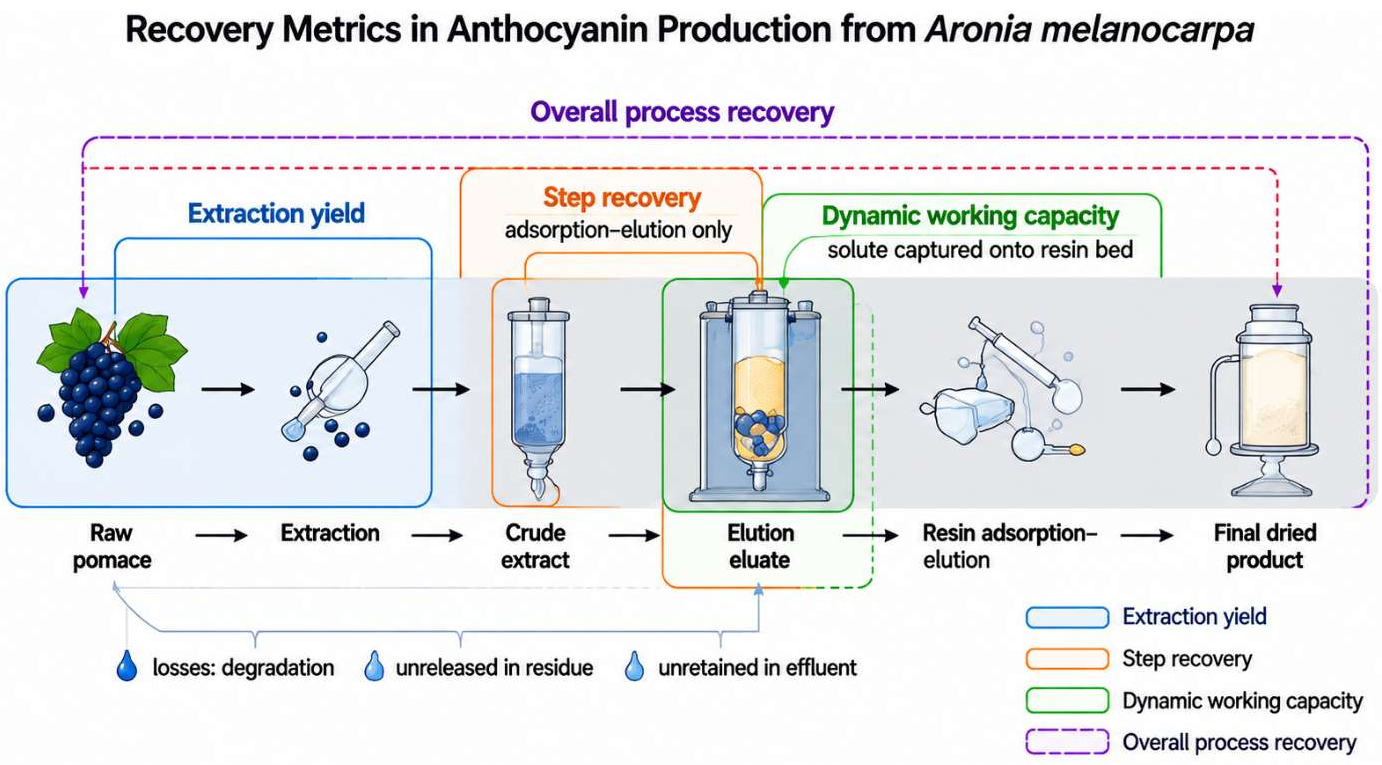


Fig. 4. Schematic illustration of distinct recovery-related metrics for anthocyanin production from Aronia melanocarpa. Schematic showing the material boundaries for extraction yield, step recovery, dynamic working capacity and overall process recovery. Note that step recovery describes only one unit-operation boundary, whereas overall recovery accounts for mass losses across the complete workflow from raw pomace to final dried product. Different denominators lead to non-comparable numerical values even under identical experimental conditions.

Figure 4 illustrating the material limits for extraction yield, step recovery, dynamic working capacity, and overall process recovery. Extraction yield primarily describes the transfer of target compounds from the feedstock into the recoverable stream. Static resin capacity reflects adsorption under equilibrium conditions; dynamic working capacity depends on the breakthrough criterion, flow rate, and bed conditions. Elution-step recovery is calculated relative to the compound mass entering that step, whereas product purity describes its mass fraction in the final dry product. These measures are not interchangeable. For process comparisons, overall recovery may be defined as the mass of analytically confirmed intact anthocyanins in the final product divided by an initial reference mass of extractable intact anthocyanins in the raw material. That reference should be established using a validated extraction method, with its operational definition stated explicitly; it need not represent the absolute total anthocyanin inventory in the feedstock. Balances should account for plant residues, circulating liquid, liquid held in the bed and pipes, effluent, eluate, and concentrated product, avoiding double-counting of recirculated material.

The approximately 96% reported by Roda-Serrat et al. [14] refers to recovery during adsorption–desorption, whereas the yields of 5.25 and 6.34 mg/g reported by Wathon et al. [12] are expressed per mass of dry pomace. Because they describe different stages and denominators, their magnitudes cannot be used to rank the corresponding complete processes. Under consistent feedstock, analytical, and product specifications, overall recovery should be reported together with retention of major glycosides, dry-basis product purity, productivity, and net solvent consumption. The suggested metrics and their boundaries are given in Table 4.

5.2. Molecular Integrity and Product Quality

Chokeberry anthocyanins consist mainly of cyanidin glycosides. In aqueous media, their structures undergo pH-dependent equilibria that affect color and absorption spectra; such reversible changes should not automatically be classified as chemical degradation. Prolonged heating, oxidation, or particular acidic treatments may also cause glycosidic hydrolysis and irreversible decomposition [3, 4]. Color changes or changes in total absorbance therefore do not establish the retention of the original glycosides. The pH differential method provides an operational measure of total monomeric anthocyanins but cannot differentiate individual glycosides. HPLC-DAD can separate and quantify principal chromatographic components, with mass spectrometry used where needed for structural confirmation [22, 23]. Matrix composition differs across processing stages and may affect analytical recovery and detector response. Calibration equivalents, wet- or dry-basis reporting, sample preparation,

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and matrix corrections should consequently be harmonized. Wathon et al. [12] reported differences in the relative amounts of glycosides and aglycone in products from batch and integrated operations. These differences may arise from glycosidic hydrolysis, resin selectivity, or differential losses among compounds. Relative percentages cannot distinguish the mechanisms without measurements of absolute component masses and their distribution across phases.

The intended application determines the appropriate separation target. Food colorants must meet requirements for color, stability, and residual solvents as well as anthocyanin content; some co-existing phenolics may also alter color through co-pigmentation. Preparation of individual reference compounds instead requires more stringent structural identification and selectivity. There is no universally optimal purity or recovery target independent of product specifications.

5.3. Resin Regeneration, Solvent Recycling, and Resource Use

Repeated exposure to crude extracts can leave phenolic residues, colloidal deposits, or mechanically damaged particles in a resin bed, potentially changing dynamic capacity, selectivity, and pressure drop. A high recovery in one adsorption–desorption cycle cannot replace multicycle stability assessment; cycle number, regeneration conditions, and changes in working capacity need to be reported.

Aqueous ethanol may be used in both extraction and elution, but eluent strength and volume affect desorption, co-elution, product concentration, and the burden of subsequent solvent recovery. Candidate elution conditions should be compared using net anthocyanin recovery and actual solvent use under equivalent product specifications.

Deep eutectic solvents can increase solute release, but dilution or solvent exchange before resin loading adds handling steps and potential losses [16, 17, 24, 25]. Studies involving other feedstocks indicate that combining such solvents with resins is feasible, but do not demonstrate that every formulation is compatible with chokeberry extracts. Engineering comparisons should record fresh solvent, water, energy, and resin use per unit mass of intact anthocyanins ultimately recovered, together with solvent-recycling efficiency, operating cycle, and scale. Techno-economic analysis and life-cycle assessment require explicit system boundaries. Without relevant resource data, neither a deep eutectic solvent nor an integrated process should be described as environmentally or economically superior on the basis of its label alone.

5.4. Scale-up and Operating Boundaries

Scale-up requires matching the upstream release rate to the dynamic processing capability of the adsorption unit. Increasing feedstock throughput does not guarantee a proportional increase in anthocyanin retention per unit time; feed concentration, circulation rate, bed residence time, and regeneration cycle must be considered together.

Table 4. Harmonized metrics for overall recovery, product quality, and operating resources.

Metric	Proposed definition and boundary	Potential misinterpretation
Raw-material-based extraction yield	mg intact anthocyanins/g dry feedstock	Not equivalent to overall recovery
Step recovery	Target mass leaving a step / target mass entering that step	Not directly comparable across steps
Dynamic working capacity	Net mass retained before a defined breakthrough criterion / dry resin mass; account for changes in bed liquid inventory	Not equivalent to static saturation capacity
Overall recovery	Mass of intact anthocyanins in final product / reference extractable intact anthocyanin mass in raw material	Reference extraction basis must be specified
Product purity and composition	Anthocyanin mass / dry product mass; distribution of major glycosides	Total phenolic purity is not anthocyanin purity
Productivity	Target-compound mass in final product / effective operating time	Whether regeneration downtime is included must be stated
Solvent intensity and resin cycling stability	Net fresh solvent / mass anthocyanins recovered; capacity change over cycles	Extraction-stage solvent use alone is insufficient

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The 50-fold scale-up reported by Vauchel et al. [11] supports the feasibility of obtaining antioxidant phenolic recovery similar to laboratory performance for the configuration studied. It does not provide sufficient evidence on long-term resin regeneration, batch-to-batch variation of the raw material, or operating cost. Short-term scale-up performance should not be presented as demonstration of stable, continuous industrial operation. Scaling a fixed bed also requires information on resin particle size, bed height, superficial velocity, empty-bed contact time, and pressure drop. Axial dispersion and flow distribution can change with equipment dimensions, making validation of laboratory model parameters against breakthrough data from larger systems necessary. Time-dependent feed composition during external recirculation presents an additional predictive challenge.

Continuous or semicontinuous operation requires coordination between extraction and adsorption–elution–regeneration cycles. Multiple columns or a buffer vessel may reduce interruptions but increase equipment and control requirements. The choice among batch, semicontinuous, and continuous operation should depend on product specifications, processing scale, and measured resource inputs. Ultimately, engineering value should be evaluated as reliable production of a specified product, rather than as a favorable extraction percentage, resin capacity, or treatment time at a single operating point.

6. KEY SCIENTIFIC QUESTIONS AND RESEARCH PRIORITIES

6.1. Sources of Integration Benefits and Concentration Feedback

Published studies show that direct integration can improve the output of chokeberry phenolics or anthocyanins under certain conditions [10, 12], but integration also alters circulation, mixing, and residence time. The central unresolved question is whether adsorption merely captures material already released or also increases net release from plant tissue through feedback mediated by liquid-phase concentration.

Figure 5 shows a proposed four-arm experimental setup. A four-arm experimental comparison is proposed: (A) conventional batch extraction with matched solvent, temperature, and raw material; (B) recirculated extraction through an empty column; (C) recirculated extraction through an inert packing selected to approximate the hydraulic behavior of the resin bed; and (D) recirculated extraction through the active adsorption resin. Comparisons among B, C, and D would help separate circulation, packing-related, and adsorptive effects. Absolute masses of major anthocyanins should be measured in the plant residue, circulating liquid, resin, effluent, and eluate. Even if D produces more final product, reduced residual anthocyanin mass in the plant material must be demonstrated before claiming additional release.

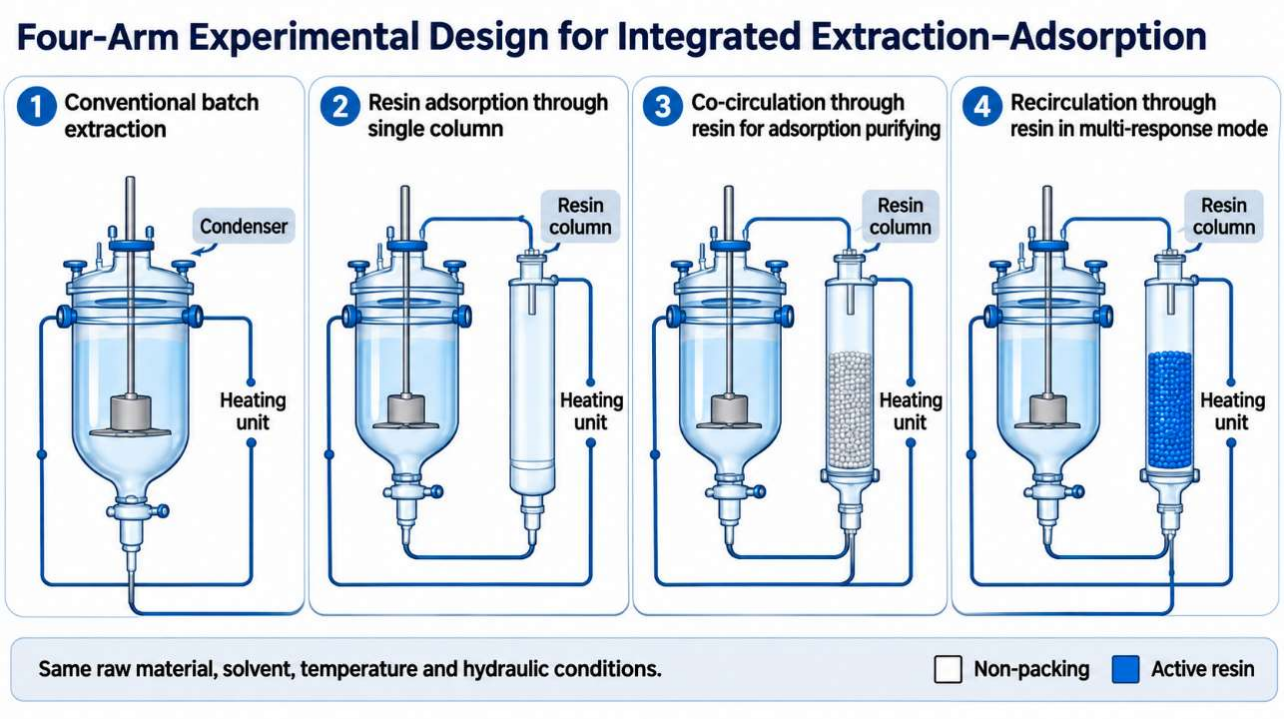


Fig. 5. Proposed four-arm experimental setup to disentangle adsorptive concentration-feedback effect from hydrodynamic mixing-related effects in integrated extraction-adsorption.Four experimental groups A-D with matched raw material, solvent, temperature and hydraulic conditions. Group A: conventional batch extraction; Group B: recirculation through empty column; Group C: recirculation through inert non-adsorptive packing; Group D: recirculation through active macroporous resin. Multiphase mass measurement points include plant residue, circulating liquid, column effluent, resin phase and eluted product.

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6.2. Multicomponent Competition and Dynamic Selectivity under Time-Varying Feed Conditions

In a coupled process, feed concentrations and component ratios evolve during extraction, and apparent resin selectivity may consequently vary over time. Differences in the release and adsorption of anthocyanin glycosides, phenolic acids, and proanthocyanidins could jointly determine final product composition.

Adsorption equilibria and dynamics for individual target glycosides and representative co-extracted phenolics could first be compared in single-solute and binary systems, followed by component-specific breakthrough and elution measurements for real extracts. The aim is to separate selective retention, competitive displacement, and chemical loss rather than to interpret every effect from changes in total anthocyanins or total phenolics.

6.3. Multiscale Model Validation and Engineering Applicability

Existing fixed-bed models describe aspects of anthocyanin adsorption–desorption [13], whereas recirculated extraction–adsorption additionally involves continuous release, changing feed composition, and increasing resin loading. Introducing more fitted parameters with insufficient observables often makes parameter identification less reliable rather than clarifying the underlying mechanisms.

The main factors that affect the final recovery yield of anthocyanins and the quality of the final product during integrated extraction-adsorption process in Aronia melanocarpa are shown in Figure 6. The fishbone diagram provides the key variables for the final result through the central stalk. This includes the feedstock characteristics such as particle size, ripeness and pretreatment methods on the top branch. The extraction process can be intensified using ultrasound. Hydrodynamic and mass transfer properties are also highlighted, this depends on the mixture characteristics, multiple stages and molecular interactions. Chemical degradation can occur due to the pH and oxygen present. The resin characteristics of endothelial resin include pore structure, adsorption mechanism, competitive adsorption, stability and lifetime. On the bottom branch, operational factors such as temperature, solvent characteristics and regeneration losses are included. Regeneration losses are due to desorption and elution conditions. This will affect the resin lifetime and overall efficiency of the process.

Key parameters should first be constrained by independent extraction, static adsorption, and fixed-bed experiments. Units should then be coupled through mass-conservation equations that include relevant phase inventories. Predictions should be tested under particle sizes, flow rates, bed heights, and feed compositions not used during calibration. Scale-up assessment also needs multicycle resin regeneration, raw-material variability, and resources consumed per unit product. Research should prioritize quantitative attribution of integration benefits, validation of component selectivity with time-dependent feeds, and definition of the operating limits within which transport models and scale-up strategies can be applied (Table 5).

7. FUTURE DIRECTIONS

Future work on Aronia melanocarpa anthocyanin extraction-adsorption should focus on optimising the process. This begins by standardising the characterisation of the feedstock and creating mass balance data for each component. This will improve experimental consistency and provide accurate data on anthocyanin recovery. Work should continue looking at optimising solvent-extraction and adsorbent-resin systems taking into account solvent compatibility, competition and stability of the individual cyanidin glycosides.

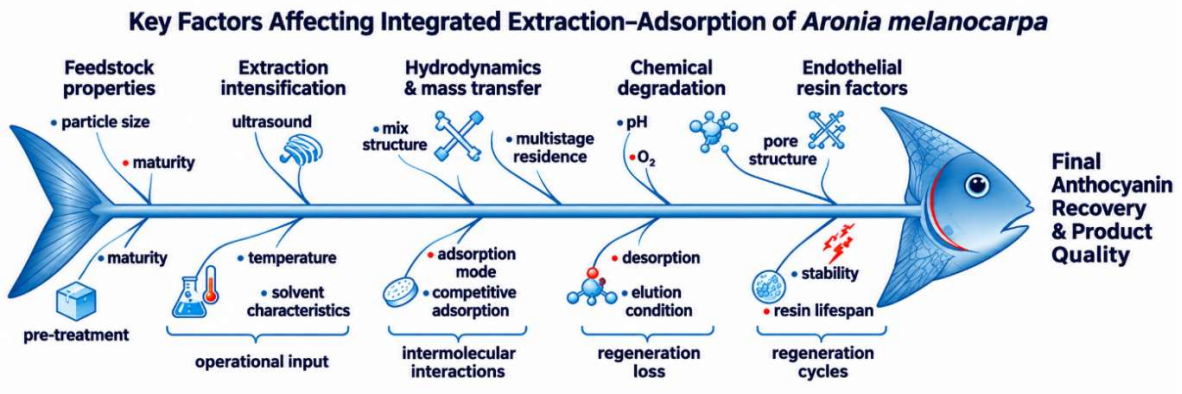


Fig. 6. Fishbone diagram summarizing major factors influencing final anthocyanin recovery and product quality in integrated extraction-adsorption of Aronia melanocarpa. Main branches include feedstock characteristics, extraction intensification parameters, adsorption-resin properties, hydrodynamics & mass-transfer, chemical degradation, and industrial practical constraints (resin regeneration, solvent consumption, scale-up). Each main branch lists representative sub-factors discussed in this review.

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Table 5. Major scientific questions, evidence gaps, and proposed validation strategies.

Scientific question	Available evidence	Evidence gap	Proposed validation	Expected outcome
Integration gains and concentration feedback	Studies [10, 12] show improved output, but not isolated feedback	No simultaneous mass inventories across all phases	Compare A: batch, B: empty column, C: inert packing, D: active resin; match hydraulics and track phase masses	Separate capture from additional release
Time-varying component selectivity	Study [12] shows compositional differences; most data report totals	Limited component-specific breakthrough and eluate data	Progress from single and binary mixtures to crude extracts; quantify breakthrough and eluates	Control of product composition
Multiscale modeling and scale-up	Study [13] models a fixed bed; study [11] reports 50-fold scale-up	Little cross-condition or multicycle validation	Independently constrain parameters; test unseen conditions, resin regeneration, and resource use	Establish model and scale-up limits

Advanced analytical techniques should be used to determine the optimum conditions using mass transfer modelling. In-process monitoring could significantly increase adsorption efficiency. In an industrial setting future work should focus on validating the process at a pilot scale and looking at long term resin regeneration, solvent recycling and product stability. Techno-economic and life cycle assessments should be included in future work to ensure the process is feasible and environmentally friendly. This will allow efficient, scalable and sustainable technologies to be developed to recover valuable anthocyanins from chokeberry by products.

8. CONCLUSIONS

Production of chokeberry anthocyanins involves release from plant tissue, changes in the liquid environment, resin adsorption–desorption, and stability during processing. Published studies show that extraction intensification and integrated extraction–adsorption can improve output or enrichment under defined conditions. Extraction yield, step recovery, product mass fraction, and overall recovery nevertheless have distinct denominators and cannot be treated as equivalent. Mechanistically, capture of already released anthocyanins is a demonstrable redistribution among phases; enhanced release induced by lowering the liquid-phase concentration remains a conditional and incompletely verified feedback mechanism. Existing direct integration and stand-alone fixed-bed studies do not yet quantify the separate contributions of release, capture, and degradation to final recovery. A three-phase mass balance provides a useful conceptual framework, but realistic recirculation systems require explicit treatment of bed holdup, incoming and outgoing streams, and other losses. Future work should combine hydrodynamically matched controls, component-resolved phase balances, and prediction under independent operating conditions using separately constrained parameters. Retention of individual anthocyanin structures, resin regeneration, and net resource consumption also need to be evaluated. Only on a shared feedstock basis and against the same product specifications can the operating boundaries and engineering value of integrated extraction–adsorption be assessed reliably.

DECLARATIONS

Ethical Approval

We affirm that this manuscript is an original work, has not been previously published, and is not currently under consideration for publication in any other journal or conference proceedings. All authors have reviewed and approved the manuscript, and the order of authorship has been mutually agreed upon.

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Conflicts of Interest

The authors declare that they have no financial or personal interests that could have influenced the research and findings presented in this paper. The authors alone are responsible for the content and writing of this article.

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Authors' contributions

All authors contributed equally to this work,

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