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THE NEW CONCEPT OF MIEX[®] RESIN DOSE CATEGORIES. SWELLING EFFECT, REACTOR STEADY-STATE BALANCE, AND NOM REMOVAL PROCESS CONTROL

The use of powdered adsorbents for water purification has many advantages and one major drawback – lack of regeneration due to difficulty in separating powdered particles. This weakness is attempted to be broken by powdered magnetic adsorbents, in particular magnetic ion exchange resins, used to remove natural organic matter (NOM) from water. In this water treatment process, NOM removal is controlled by the adsorbent content in the reactor (adsorbent dose) and the degree of its saturation. The control over the dose and saturation is done by mutual relations between the regenerated resin stream directed to the reactor and the saturated resin stream received from the reactor. An obstacle in balancing these streams is a variable volume of the adsorbent resulting from its varied swelling, depending on the features of the solution and saturation of the adsorbent. For this reason, it was proposed to distinguish new resin dose and content categories adequate to these changes, the use of which allows full control of both streams. Thus, the reactor feed stream was associated with relative fresh resin content (*RRC*) and relative fresh resin dose (*RRD*), which indicate the volume occupied by the regenerated adsorbent in the solution of water during purification. However, the stream received from the reactor was associated with saturated resin content (*SRC*) and saturated resin dose (*SRD*), which indicate the volume occupied by saturated adsorbent in the solution of water under treatment. In turn, these two categories of contents/doses are related to the swelling degree (η_{SR}). Another role was assigned to the third dose category, which is absolute fresh resin dose (*ARD*), referring to the volume occupied by the regenerated adsorbent in the solution of demineralized water. Thanks to two key features with reference properties (demineralized water, regenerated adsorbent), *ARD* allows one to transfer laboratory results to practice and to compare the results of various research. The resin loss factor described by the η_{LS} indicator was also included in this structure.

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ABBREVIATIONS

Absolute fresh resin dose	–	<i>ARD</i>
Bed volume(s)	–	<i>BV</i>
Dual-stage (type of reactor)	–	<i>DS</i>
Effective resin dose	–	<i>EjRD</i>
Equivalent (effective) resin dose	–	<i>ERD</i>
Fluidized ion exchange (treatment process and reactor type)	–	<i>FIX</i>
High-rate (type of reactor)	–	<i>HR</i>
High-rate counter current (type of reactor)	–	<i>HR-CC</i>
Magnetic ion exchange resin	–	<i>MIEX[®]</i>
Natural organic matter	–	<i>NOM</i>
Relative fresh (regenerated) resin content	–	<i>RRC</i>
Relative fresh (regenerated) resin dose	–	<i>RRD</i>
Regenerated resin volume	–	<i>RRV</i>
Suspended ion exchange (treatment process and reactor type)	–	<i>SIX[®]</i>
Saturated resin content	–	<i>SRC</i>
Saturated resin dose	–	<i>SRD</i>
Saturated resin volume	–	<i>SRV</i>

SYMBOLS

Swelling degree	–	η
Swelling degree between RRD and ARD	–	η_{RA}
Swelling degree between SRD and ARD	–	η_{SA}
Swelling degree between SRD and RRD	–	η_{SR}
Resin loss index	–	η_{LS}
Settled resin volume unit	–	L_r
Resin suspension volume unit	–	L_s
Regenerated resin suspension flowrate	–	q_{RR}
Saturated resin suspension flowrate	–	q_{SR}
Settled resin volume	–	V_r
Resin suspension volume	–	V_s

1. POWDERED ADSORBENTS

The adsorption process plays a special role in the water treatment train due to the ability to remove solute molecules. The main features of adsorbents that determine their attractiveness include the adsorption capacity (maximum mass of bonded solution components per mass unit of adsorbent) and the adsorption rate (mass of solution components bound by unit mass of adsorbent per unit of time). The capacity is proportional to the internal surface area of the adsorbent, hence the desire to maximize it. Regardless of the value of the internal surface area, diffusion into the inside of the adsorbent grain takes place through the external surface of the grain, which is relatively larger for smaller

grains. Thus, the particulate adsorption material will have faster adsorption kinetics than the same material with larger grains. This allows comparable technological effects in reactors with significantly smaller volumes. However, the reaction rate decreases as the adsorption capacity saturation is depleted, which may undermine the sense of their use. To minimize this effect, the powdered adsorbents are only saturated to a limited extent, after which they are wasted or regenerated. The first solution is not very rational. The second one requires effective separation of the adsorbent from the liquid. Isolating powdered particles in pure form is almost impossible, as long as they are not capable of agglomeration.

Combining powdered particles into larger structures requires the activation of attractive forces between them. Such opportunities are offered, among others, by magnetic interactions. In recent decades, many powdered adsorbents with magnetic properties have been created, including ion-exchange resins [1–4], polymeric adsorbents [5, 6], activated carbons [7–10], nanoadsorbents [11, 12], composites of various adsorbents [13, 14].

So far, the most widely used powdered magnetic adsorbents used in practice are MIEX[®] ion exchange resins, in the MIEX[®]DOC, MIEX[®]Gold and MIEX[®]Plus varieties. The first two are strong base anion exchange resins used to remove natural organic matter (NOM) from water. The first being in use for over 20 years, has been thoroughly tested [15, 16]. Information on the other (newer) one has appeared just in a few reports as yet [4, 17–22].

The concepts contained in this work are based on experiments with MIEX[®]DOC resin, equally useful for all magnetic powdered adsorbents, having swelling capacity, and used in moving bed reactors.

2. POWDERED RESIN REACTOR

The NOM removal process using ion exchange magnetic resins uses different reactor variants, such as dual-stage reactor (DS) [23], high-rate reactor (HR) [24] and its high-rate counter current version (HR-CC) [25], fluidized ion exchange (FIX) [26], or suspended ion exchange reactor (SIX[®]) using mechanical mixing [27] or a hydraulic labyrinth mixing [28].

A common feature of all design solutions of these reactors is the adsorbent movement, which circulates between the reactor module (contact of the adsorbent with purified water) and the regeneration module (contact of the adsorbent with the regenerating agent) (Fig. 1). One of the key tools for controlling the NOM removal process in such a reactor system is the content of adsorbent in the reactor module. Under steady-state conditions, this content is constant, which means that the removed amount (mass) of saturated resin must equal the amount (mass) of regenerated resin supplied. If the composition and properties of the feed water remain relatively stable, then with the set contact time value also the effects of purification and saturation of the adsorbent do not

change. Changing the process conditions can be obtained by temporarily unbalancing the amount of resin delivered and received. Then its saturation decreases (when supply prevails over collection) or increases (when supply is less than collection). Decreasing saturation increases the efficiency of NOM removal, while increasing saturation reduces it. In any case, control and balancing of the resin cycle form the basis for supervising the reactor's operation.

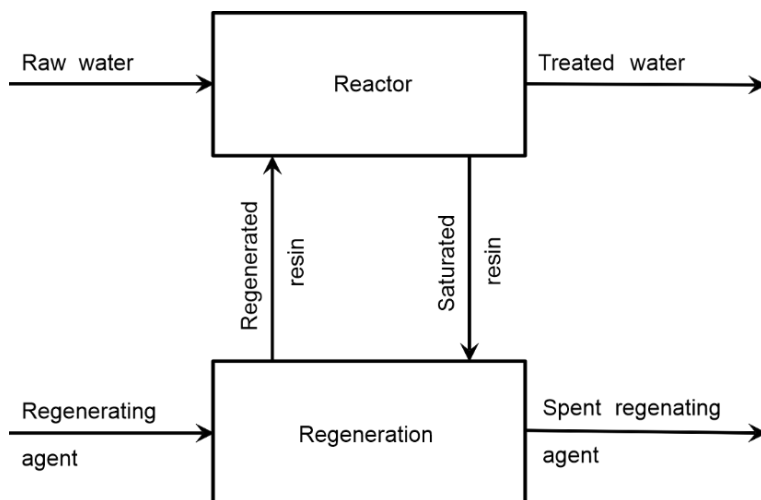


Fig. 1. General operating diagram of the continuous anion exchange process carried out with the use of powdered magnetic adsorbent

The reactor hydraulic system with magnetic powdered adsorbent consists of tanks, pipes connecting them as well as pumps and valves directing individual streams according to their purpose. Depending on the type of reactor, there are two or three different adsorbent streams in the system. Two of them always occur and relate to the transport of saturated resin from the reactor to regeneration and the return transport of regenerated resin to the reactor. In addition, in the DS reactor system, there is resin circulation between a settler and the reactor, and in the HR-CC system, the resin is displaced between two stages of HR reactors containing an adsorbent of different saturation. In each of these systems, adsorbent circulation control is carried out on a volume basis. This means that volume flowrates (with continuous flow) or batch volumes (with periodic flow) of the adsorbent exchanged between the reactor and regeneration modules must have such selected values that they correspond to the same quantities (masses) of the adsorbent. However, individual streams differ not only in the content of the adsorbent but also in its saturation and the properties of the solution with which it has contact. These three factors must be taken into account when mass balancing of the powdered adsorbent cycle is carried out on a volume basis.

3. SWELLING EFFECT

Solvent swelling is a feature of many natural and synthetic materials. Polymeric ion-exchange resins swell on contact with water as a result of the solvation of functional groups and the tendency to reach equilibrium between internal and external ionic solutions. The swelling degree depends on the structure of the ion exchanger, the strength of bonds in its structure, and the physical and chemical properties of the solution with which it has contact, such as ionic strength and pH of the solution, temperature, and type of exchanging ions [29, 30]. The presence of an internal ionic solution allows the penetration of solute molecules into the internal structure of the ion exchanger and strongly influences the kinetics and thermodynamics of the exchange reaction [31].

It has been proven that the solution type used to prepare the MIEX[®]DOC resin suspension affects the ionite volume as a result of variations in its swelling [32]. The theses of this study indicate that: (i) maintaining a constant measure of resin dose requires reference of the adsorbent's volume to its suspension in demineralized water, (ii) fresh or regenerated resin in the solution of purified water shows swelling increasing its volume concerning the volume of the same portion of adsorbent in demineralized water, (iii) saturated resin in contact with purified water shows swelling increasing in its volume relative to the volume of the same portion of fresh adsorbent in purified water, (iv) swelling degree of the resin increases with its progressive saturation, up to a certain limit value, which is reached under relatively low adsorbent load conditions (relatively low bed volumes, *BV*).

On their basis, two practical conclusions can be formulated: (i) natural waters tested have unique chemical properties, difficult to fully describe and model, therefore only measuring the volume of ion exchanger in demineralized water allows comparing the adsorbent doses used in various studies and applications, (ii) the same portion of resin will show different volumes depending on the conditions in which it is located, i.e., location in the reactor system and maintaining the balance of adsorbent circulation in the system requires taking these differences into account.

One of the possibilities to implement these postulates is to distinguish several categories of adsorbent doses, depending on the swelling degree, in place of one dose category used so far, which ignores the impact of this phenomenon.

4. RESIN DOSE CATEGORIES

In addition to the general concept of resin dose, understood as the volume of the sedimented (to constant volume) portion of the ion exchanger contained in a specific volume of liquid, several other dose concepts have appeared in the literature that are relevant to the conduct of the process. Boyer and Singer [33] popularized the concept of effective resin dose (*ERD*), understood as a volume of regenerated resin which is a process equivalent to a specific volume of saturated resin. Due to the key aspect of the

equivalence of these volumes (doses) of resin, a better term for the dose proposed by Boyer and Singer is the equivalent dose. Another work [34] proposed the concept of effective resin dose (*EfRD*) as the lowest dose of regenerated resin, the increase of which does not improve NOM removal. The minimum resin usage proposed by Qi et al. [35] has similar significance.

When considering the use of powdered magnetic resin in a water purification system, a distinction between the concepts of content and dose of resin should be made. The content has a more general sense and applies to every element of the resin-containing system, in particular to the reactor, settler, saturated resin tank, regenerated resin tank, and streams connecting these devices (Fig. 2). In turn, the dose corresponds to the resin content in the mass transfer zone, i.e., in the reactor. It is a special case of this content of adsorbent, which is involved in the ion exchange reaction in contact with water under treatment. It is the same as the dose of adsorbent used in the laboratory jar test.

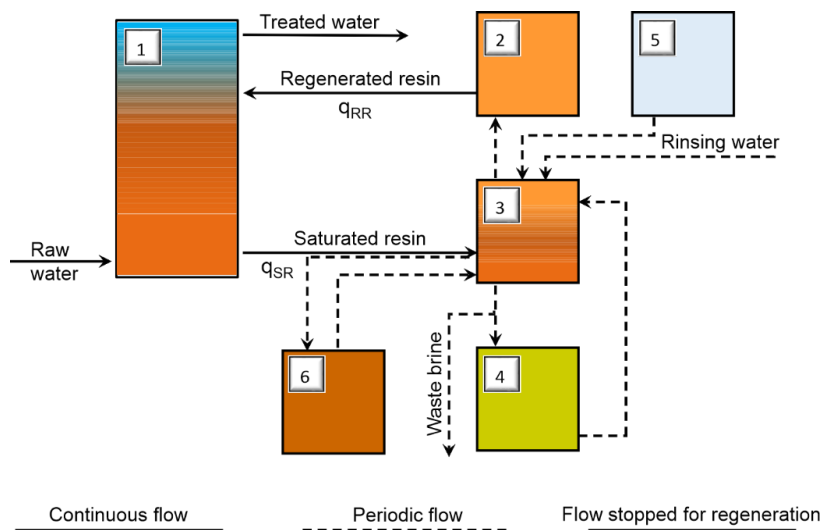


Fig. 2. Process diagram of the HR reactor operated in the variant of continuous resin exchange and batch regeneration: 1 – reactor, 2 – fresh (regenerated) resin tank, 3 – regeneration tank, 4 – used brine tank, 5 – fresh brine tank, 6 – temporary (for regeneration time) saturated resin detention tank

Balancing the content of powdered resin in the reactor system requires determining the amount (solid material) of the saturated adsorbent taken from the reactor and the amount (solid material) of the regenerated adsorbent supplied to the reactor (Figs. 1, 2). In the hydraulic system, these amounts are evaluated by the wet adsorbent volume. The volume measure of the resin content is the ratio of the adsorbent volume (V_r) to the volume of its aqueous suspension (V_s). Hence the maximum possible resin content is $1 L_r/L_s$. Usually, the highest content of adsorbent occurs in the regeneration module.

This minimizes the necessary tank volumes. In the reactors themselves, the resin content can vary considerably. And so, in the DS reactor, it represents a few percent, and in the HR reactor several dozen percent of the maximum value. The ratio of mass to volume of resin is not constant due to its swelling.

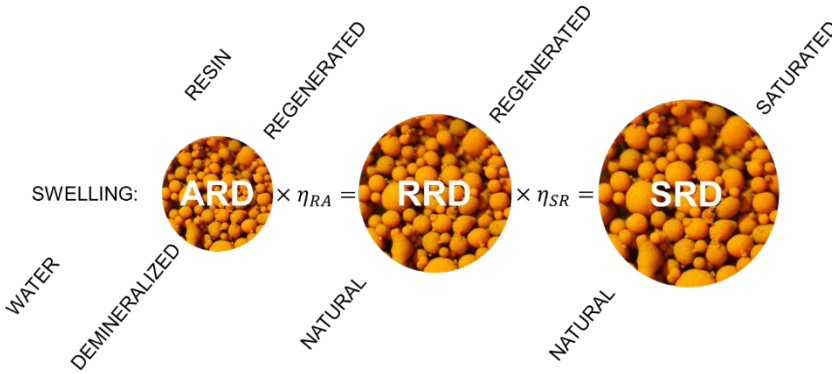


Fig. 3. Graphical demonstration of the key resin dose categories equal by mass of solid material but characterized by the volume differences caused by the swelling effect and related to the swelling degree indicators (η_{RA} , η_{SR})

Magnetic powdered resins are always used in the form of aqueous suspension. Therefore, their volume in a dry state is not determined. In a water suspension, a fixed portion of regenerated resin shows the smallest volume when in contact with demineralized water, then increasing in contact with natural water and with increasing adsorbent saturation [32]. Sorting out the relationship between changing resin portion volumes requires the introduction of new categories of powdered adsorbent content and dose. Due to the specificity of magnetic powder adsorbents, it was proposed to distinguish saturated resin (*SRD*) and regenerated resin doses (Fig. 3). In the regenerated adsorbent range, a relative fresh resin dose (*RRD*), which corresponds to the portion of the regenerated resin contained in the purified water solution, and an absolute fresh resin dose (*ARD*), which corresponds to the portion of the regenerated resin contained in demineralized water were distinguished. If the three proposed doses are equivalent (correspond to the same amount of adsorbent) then they remain in a relation related to the swelling degree of the resin ($\eta > 1$):

$$RRD = \eta_{RA} ARD \quad (1)$$

$$SRD = \eta_{SR} RRD \quad (2)$$

$$SRD = \eta_{SA} ARD \quad (3)$$

where

$$\eta_{SA} = \eta_{RA} \eta_{SR} \quad (4)$$

$$SRD = \eta_{RA} \eta_{SR} ARD \quad (5)$$

If during the start-up of the reactor, a regenerated adsorbent was placed in it at a dose of RRD , and after a certain time of operation the dose value of the saturated adsorbent SRD was measured and both doses remained in relation (2), the real dose value did not change, and the reactor works sustainably, maintaining steady-state adsorbent exchange between the reactor module and the regeneration module. If the measurement results do not meet equation (2), the balance of resin exchange between the two modules is disturbed. Then the SRD measurement will indicate the value and direction of changes (increase or decrease of the regenerated resin stream directed to the reactor) required to restore the equilibrium. Assuming that the SRD measurement showed a value less than the proper value for the state of equilibrium ($\eta_{SR}RRD$), the reactor has a saturated resin deficit (ΔSRD), which can be included in the balance:

$$SRD = \eta_{SR}RRD - \Delta SRD \quad (6)$$

The restoration of equilibrium requires directing a portion of regenerated adsorbent (ΔRRD) to the reactor, the volume of which is:

$$\Delta RRD = \frac{\Delta SRD}{\eta_{SR}} \quad (7)$$

or retaining in the reactor an additional portion of ΔSRD saturated resin. Choosing one of these two options is not indifferent to the technological effect achieved. Introducing a portion of regenerated resin into the reactor will reduce the resin age and bed volume, which could potentially result in higher NOM removal. The effect would be counter-productive if a portion of saturated resin was retained in the reactor.

In practice, the correction described by eqs. (6) and (7) would be implemented by an appropriate deviation from the balance of adsorbent circulation between the reactor module and the regeneration module. Taking into account the swelling effect and the adsorbent content in the exchange streams, i.e., relative fresh resin content (RRC) and saturated resin content (SRC), the equilibrium of this system (Fig. 2) is described by the equation:

$$q_{RR} \eta_{SR} RRC = q_{SR} SRC \quad (8)$$

in which q_{RR} and q_{SR} are regenerated resin suspension flowrate and saturated resin suspension flowrate, respectively.

This example illustrates the difference in understanding the concepts of adsorbent dose and content. RRD corresponds to the real (at start-up) or equivalent (in current operation) content of the regenerated adsorbent in the reactor, while RRC is the real resin content in the regenerated adsorbent tank and the regenerated resin stream directed to the reactor (8). RRC values are greater than RRD values, and this difference is particularly expressed in the case of the DS system reactor. Large RRC values allow to limit of the volume of tanks in the regeneration module and the stream value q_{RR} . SRD corresponds to the content of saturated adsorbent in the reactor, while the SRC value may be consistent with it where saturated resin is transported directly from the reactor to regeneration (e.g., HR type reactor) or not if the saturated resin is directed for regeneration via the settler (DS reactor). In the second case $SRC > SRD$.

In the magnetic powdered adsorbent balance, a resin loss factor needs also be considered. The resin loss index ($\eta_{LS} < 1$) can be defined as the mass of adsorbent received from the reactor relative to the mass of adsorbent delivered to the reactor. Taking into account resin losses leads to the adsorbent exchange balance:

$$q_{RR}\eta_{SR}RRC = q_{SR}\eta_{LS}SRC \quad (9)$$

According to eq. (9), the stream q_{RR} balances the return stream q_{SR} associated with the reduced content of saturated adsorbent $\eta_{LS}SRC$, therefore it does not compensate for the adsorbent losses. A measurement of the SRC in the return stream will show the value $\eta_{LS}SRC$.

Table 1

Examples of individual swelling levels determined in laboratory conditions [32]

Resin	Water	Resin bed volume (BV) [m ³ /m ³]	Swelling degree
Regenerated	demineralized	0	1.0
	model water (NOM + NaHCO ₃)	<50	$\eta_{RA} = 1.034$
Saturated	model water (NOM + NaHCO ₃)	200	$\eta_{SA} = 1.100$ $\eta_{SR} = 1.064$
		≥400	$\eta_{SA} = 1.150$ $\eta_{SR} = 1.112$

The resin loss factor means that, as in the case of the η_{RA} indicator, also determination of the swelling ratio η_{SR} should be carried out in laboratory conditions, with the adsorbent losses elimination (Table 1). Determining the value of this indicator in operating conditions would lead to the determination of the resultant changes in resin volume resulting from swelling and adsorbent losses (η_{SR}, η_{RA}). The distinction between these two effects allows for simultaneous compensation of differences resulting from swelling and losses. However, one should be aware of the scale of impact of both phenomena.

Weighing the impact of swelling and losses on the equilibrium conditions of the adsorbent cycle, it can be concluded that while the corrections resulting from swelling reach a dozen or so percent (Table 1), the value of the correction related to losses should not exceed 1% ($0.99 < \eta_{LS} < 1$). Due to the relevantly lower significance of losses, this impact can be equalized periodically and omitted in the current balancing of adsorbent streams (9). Then in everyday operation conditions, it is assumed that $\eta_{LS}SRC \approx SRC$, as in eq. (8). In addition, due to the multitude of factors affecting the degree of swelling, the values of this indicator ($\eta_{SR}\eta_{RA}$) should be determined individually for each application and periodically verified. For the same reasons, the *ARD* indicator has a special role, which is the only way to compare adsorbent doses used for waters with different properties and operational parameters of the process, and, in particular, for different tests carried out by different teams of researchers.

The above description refers to one of the possible concepts of the regeneration system based on continuous adsorbent exchange and batch regeneration. An analogous approach can be used for other concepts, including the concept of batch resin exchange and batch regeneration carried out every *n*-exchange, the batch resin exchange and batch regeneration carried out at each exchange, and continuous resin exchange and continuous regeneration. Especially the latter variant is a prospective field of research.

In addition to the variants of continuous operation of the reactor described here, its batch mode is also possible. This is the only case where it is not necessary to take into account the swelling effect and related proposed categories of adsorbent dose and content. In the batch reactor, the reactor is fed once with a portion of regenerated adsorbent, which can be defined as regenerated resin volume (*RRV*). The same portion of adsorbent returns to the reactor after regeneration. If this volume has been reduced as a result of losses, it should be supplemented to its original value. However, also in this case, the swelling degree can be used to determine the value of losses before regeneration

$$\eta_{LS} = \frac{\eta_{LS}SRV}{\eta_{SR}RRV} \quad (10)$$

where $\eta_{LS}SRV$ is the result of the measurement of saturated resin volume *SRV* withdrawn from the reactor and reduced by the value of adsorbent losses η_{LS} .

5. CONCLUSIONS

The powdered magnetic adsorbent circulation between the reactor and regeneration zones provides the continuous adsorption system with extensive regulatory possibilities for removing adsorption substrates, in particular, NOM. One of the problems of balancing the adsorbent cycle is the variability of the adsorbent portion volume depending on the characteristics of the solution with which it contacts and its (adsorbent) saturation

state. This effect can be included in the balance of adsorbent circulation by using new categories of adsorbent dose and content. In this context, three states of the adsorbent/solution system and related dose categories were distinguished: (i) regenerated resin in demineralized water (*ARD*), (ii) regenerated resin in natural water (*RRD*), and (iii) saturated resin in natural water (*SRD*). New resin content categories (*RRC*, *SRC*) were defined analogously. Individual doses and contents were related by appropriate swelling indicators (η_{RA} , η_{SR} , and $\eta_{SA} = \eta_{RA}\eta_{SR}$), whose values must be determined individually, for each water source.

The proposed categories of adsorbent dose and content related to the contact of the adsorbent with natural water (*RRD*, *RRC*, *SRD*, *SRC*) will be useful in controlling the process carried out on a technical scale. The absolute fresh resin dose (*ARD*) in demineralized water has a special role to play in transferring laboratory results to practice and comparing the results of various tests.

The presented structure of adsorbent dose and content categories and their interrelationships are based on experiments with MIEX[®]DOC resin but equally useful for all magnetic powdered adsorbents, having swelling capacity and used in moving bed reactors. It is hoped that this powdered adsorbent dose categories proposal will enable readers to have a further understanding of the continuous ion-exchange process performance and will be useful for the operation of full-scale reactors and research.

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