Recovery of phosphate ores in the modified three-product column (3PC) flotation cell

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A B S T R A C T

This work details the results obtained for the rougher flotation of phosphates in a modified flotation cell known as a three-product column (3PC), at both the laboratory and pilot plant scales. Results were compared to a conventional column cell—CCC. The 3PC cell separates the drained (rejected) particles from the froth zone (third product) and uses a secondary wash water system between the feed and the froth zone (II). Bench-scale studies measured the effect of the two water surface rates on mass and metallurgical recoveries and concentrate grades (P2O5, Fe2O3, and SiO2) in all the flows. At pilot scale, the influence of wash water (Jw2) and column design on the flotation separation parameters was studied. Results showed that, compared with the conventional column cell (CCC), the 3PC yielded, in all cases, clean high-grade concentrates, with a minor concentration of impurities (Fe2O3 and SiO2). Concentrate recoveries ranged from 40% to 70% for apatite and were lower when compared to CCC, but it is believed that the third product could be recycled to the feed. This drop-back product operating with Jw2 = 0.0 cm s⁻¹ might yield 5–10% extra in apatite recoveries and enhancing this Jw2 values, the apatite recovery decreased by 0.5–3% but rejection of impurities was very high. Results appear to show that the 3PC may be used as a rougher-flash or Cleaner unit with an optional recycle of the third product into the rougher or simply discarding it. Data on the influence of some cell design and gas dispersion parameters on process efficiency are reported, and the potential practical applications for this type of cell are envisaged.

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1. Introduction

The 3PC (three-product column) (concentrate, drop-back, and tailings) cell is a modified flotation (Fig. 1) column which was originally employed by Falutsu and Dobby (1989) to determine and characterize the material that returns from the froth zone to the collection zone as drained material (dropback). Dobby (1984) and Rubinstein (1995) described a column with a similar design, but without specifying results. Gallegos-Acevedo et al. (2007) also used an apparatus similar to that described by Falutsu and Dobby to determine the loading of air bubbles in the froth phase, thereby estimating the flow of solids in the concentrate. Ata et al. (2002), with similar equipment, evaluated the collection of hydrophobic particles in the froth phase introduced into the column through the wash water.

The 3PC used in this work has been widely reported in several publications, studies, and projects (Table 1). This 3PC cell behaves similar to a classification column and its efficiency depends on particle hydrophobicity, liberation, and froth recovery. Medium-sized hydrophobic particles having high flotation rates are transferred rapidly to the concentrate, resisting froth drainage. Middlings, depending on contact angle, are either collected by bubbles or drained as drop-back material jointly with entrained or entrapped gangue slimes (Rubio, 1996; Valderrama and Rubio, 2008; Valderrama et al., 2011).

Such behavior results in obtaining rich concentrates, which is explained because in a normal straight column some material always keeps circulating: a fraction of the froth drop-back product remains at the pulp-froth interface, then returns to the froth and another reports to the tailings. The fraction returning to the froth is again rejected, and a new cycle starts. This material is usually composed of low-grade middling and/or entrained gangue slimes, and since it has to leave the column it may either contaminate the concentrate or be transferred to the tailings, causing losses, should the amount of valuable in middlings be high. In
conventional columns, the presence of low-grade drop-back material limits the cell enrichment but recoveries are usually high because of the middlings being reported to concentrates.

Thus, in the 3PC column, the collection zone becomes free from froth-drained by-products and their circulation and remains constant in terms of physical properties (pulp density, viscosity), solid content, ore grades, pressure on the sparger, and holdup values (Rubio and López, 1992).

Due to the separation selectivity, this 3PC cell may be used in various forms and it has been found that independently of feed quality, enrichment will depend on the amount of liberated hydrophobic particles in the system. This makes this flotation cell amenable and efficient in the treatment of wasted, low-grade tailings material; as a rougher-flash flotation of copper (Santander et al., 1994); as a cleaner of a copper rougher feed (Rubio, 1996; Valderrama et al., 2011); as a rougher-cleaner gold (Rubio, 1996); as a cleaner of Zn–Pb rougher concentrates (Cabral and Rubio, 1996); as a rougher-cleaner of fluorspar (Rubio and López, 1992); as a rougher-cleaner of gold (Valderrama and Rubio, 2008); and for tailing treatment.

The objective of this study was to validate the 3PC technology, in the flotation of feed flotation phosphate fine ore at both laboratory and pilot scales, comparing grade flotation concentrates with a CCC (conventional column cell). A brief characterization of some gas dispersion values in this column was also performed.

2. Experimental

2.1. Materials

2.1.1. Ores

A phosphate ore sample, used throughout the studies, was obtained from the concentrate plant at Araxá (Vale Fertilizers-Southeast Brazil) by collecting it from a by-pass pipe that takes the underflow material from the desliming to the conditioning tank with depressant and pH adjustment.

Table 2 shows the levels of the main elements that constitute the sample.

2.1.2. Reagents

To determine the distribution of bubble size and hold-up in the collection zone of the column, solutions of Dowfroth 250 (DF 250 – CH3 (OH)2OH, PM = 264.37) were prepared in order to reduce the superficial tension in the liquid/air interface.

For flotation studies, soybean oil soap was used as the collector of phosphorus-bearing minerals, obtained after saponification of the soybean oil at a temperature of 70°C for a period of 15 min with an Oil/NaOH ratio of 7:1. Gelatinized corn starch was used as gangue mineral depressant with a Starch/NaOH ratio of 4:1, with 10 min conditioning time at 20% solids by weight. Both reagents were prepared at 1% concentration with distilled water. Both reagents were prepared with a 1% concentration of distilled water. For pH adjustment, a 10% NaOH solution was used. The water supply from the city of Porto Alegre (laboratory) and industrial water for the pilot scale studies were used to attain the correct percentage of pulp solids.

2.2. Methods

2.2.1. Holdup measurements

Measurements were done for holdup that was apparent in the collection zone of the column for a two-phase system (air/liquid) using a conventional column cell (CCC). The technique used, as described by Finch and Dobby (1990), consists of determining the pressure difference between two points over a section of length equal to 83 cm. Pressure was sensed by water-filled manometers (U-tube manometer) placed laterally against the column; the difference in fluid height in a liquid column manometer is proportional to the pressure difference.

Thus, the holdup is determined by the following equation:

\[ \hat{v}_g = \Delta H/L, \]

where \( \Delta H \) is the difference in pressure read in the manometers, and \( L \) is the distance between the measurement points.

Solutions of DF 250 with water from Porto Alegre (South Brazil) were prepared at the desired concentrations in a 30 L tank under stirring at 550 rpm. After 10 min of conditioning, the solution was led to the column at 1.37 m from the bottom and the tailings were extracted from the bottom of the column. Air was injected at the base of the column through a porous stainless steel tube with pores of 5 μm in diameter.

![Fig. 1. Modified three-product column (3PC) flotation cell. (1) Collection zone: located between the bubbler and the ore feed point; (2) secondary washing zone: between the feed and the point addition of the wash water 2; (3) inflection zone: located between the point of addition of wash water 2 and upper tube (froth and dropback-4); (4) cleaning zone: between the top of the column and the upper end of the tube 2; and (5) zone of the drained – froth rejected particles: between the inflection point until the end of the tube 5.](image)
The measurements were done by dynamic testing, where the feed flows ($J_L = 0.66 \text{ cm s}^{-1}$) and tailings were controlled to maintain a constant flow rate and volume within the column.

The effect of superficial gas velocity for values of 0.33, 0.49, 0.66, 0.82, and 0.99 cm s$^{-1}$ and a concentration of DF 250 (5, 10, 20, 30, and 40 mg L$^{-1}$) was evaluated against the apparent holdup as a function of the superficial gas velocity for DF 250 concentrations. The addition of the surfactant reduces the average bubble diameter and the air holdup increases with superficial gas velocity.

### Table 2

<table>
<thead>
<tr>
<th>Grades, %</th>
<th>$P_2O_5$</th>
<th>CaO</th>
<th>Fe$_2O_3$</th>
<th>SiO$_2$</th>
<th>Al$_2O_3$</th>
<th>MgO</th>
<th>BaSO$_4$</th>
<th>TiO$_2$</th>
<th>Nb$_2O_5$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>13.0</td>
<td>12.0</td>
<td>30.2</td>
<td>15.9</td>
<td>4.8</td>
<td>1.2</td>
<td>0.7</td>
<td>5.8</td>
<td>0.5</td>
</tr>
<tr>
<td>Standard deviation</td>
<td>0.35</td>
<td>0.08</td>
<td>0.37</td>
<td>0.07</td>
<td>0.09</td>
<td>0.05</td>
<td>0.06</td>
<td>0.03</td>
<td>0.02</td>
</tr>
</tbody>
</table>

The LTM-BSizer technique (Rodrigues and Rubio, 2003; Rodrigues et al., 2004; Rodrigues, 2004) was used to determine the bubble-size distribution generated by the column bubbling (porous stainless steel cylinder of 40 mm diameter, 10 cm height, and 5 µm pore diameter). This technique is based on the capture concept in which images are obtained of the stabilized bubbles from a horizontal angle.

The bubbler used to generate bubbles in the laboratory CCC/3PC units was coupled to the base of an acrylic column that was 200 cm high and 72 mm in diameter. The sample for the determination of the bubble size was taken at 80 cm above the point where the bubbles were generated. At this point, a partial flow was conducted up to the cell where the bubbles were photographed. To obtain photographs, the flow was interrupted and the bubbles were trapped in the cell in contact with the upper part (glass plate) of it (Rodrigues et al., 2004). The images were then transferred to the computer and processed with the software Image Tool (version 3.00) to determine the average bubble-sized distribution ($d_{12}$, Sauter). The effect of the concentration of DF 250 (ranging from 5 to 40 mg L$^{-1}$) was evaluated for a superficial air velocity of 0.49 cm s$^{-1}$.

### 2.2.3. Pilot plant scale (4th column).

The CCC flotation trials were performed in a column with an internal diameter of 9.0 cm, a height of 7.6 m, and an effective volume of approximately 48 L, composed of acrylic modules joined by flanges.

The pulp used was sampled via a bypass in the piping, taking the underflow material from the cyclone (40–44% of solids by weight) to the conditioning tank. The pulp was conditioned with gelatinized corn starch as the depressant, pH equal to 12, and soybean oil soap. After conditioning, the pulp was diluted to 25% of solids by weight and fed at 1.26 m from the top of the column. Bubbles were generated with controlled pressure and flow via a forced air passage in a porous pipe at the base of the column. The pulp/froth interface was controlled by a level sensor that was attached to the tailings pump. After reaching the stationary stage, product samples were collected, flocculated, and dried in an oven for 24 h at 80°C. Then, the samples were weighed, disaggregated, and separated in aliquots for chemical analysis by X-ray fluorescence spectroscopy.

The flotation studies in the 3PC followed the procedure adopted in the CCC studies. For the 3PC, the drained product was obtained from the froth phase rejects. The adaptation of the CCC to the 3PC was done by replacing the last part of the CCC (1.50 m) with the section containing the inflection zone. After reaching the stationary stage of the system, samples of all products were measured to determine the mass and metallurgical balance. The effect of superficial velocity of wash water II ($J_{W2}$) in the 3PC, on mass and metallurgical recovery of apatite, $P_2O_5$ concentrate grades, and impurities (Fe$_2O_3$ and SiO$_2$) contents was measured.

### 3. Results and discussion

#### 3.1. Gas dispersion parameters

Fig. 2(a) shows the holdup apparent in the collection zone of the column as a function of the DF 250 concentration for a superficial air velocity of 0.49 cm s$^{-1}$. Fig. 2(b) shows a variation of the gas holdup as a function of the superficial gas velocity for DF 250 concentrations from 5 to 40 mg L$^{-1}$.

For all the tested concentrations of DF 250, the holdup displayed a linear variation with an increase in superficial gas velocity. Moreover, an increase was observed in the holdup as the concentrations of DF 250 increased. This fact can be explained by the decrease in the size of bubbles generated by the bubbler with an increase in the surfactant concentration, which results in the bubbles rising more slowly and causing an increase in gas holdup (El-Shall et al., 2000; Finch and Dobby, 1990).

Table 3 presents the results of average bubble diameter and bubble surface area flux (SB) resulting from an increase in the DF 250 concentration. The addition of the surfactant reduces the average bubble diameter and, consequently, there is an increase in the SB. In these experiments, the SB ranged between 28 and 50 s$^{-1}$, which are typical values for an air/water two-phase system. In the collection zone, the air holdup increases with superficial gas
velocity, solid loading in the bubbles, and pulp velocity (Uribe-Salas et al., 2003; Yianatos, 2002; Dobby and Finch, 1986). In the froth zone, the air holdup increases with the size of the bubbles and froth height, and decreases with an increase in the air flow and wash water (Yianatos, 2002; Finch et al., 1989).

In analyzing data from the literature for measurements of Sb and $J_g$ in laboratory and pilot columns, and laboratory-and pilot-scale mechanical cells, Finch et al. (2000) showed that there is a linear relationship between the gas holdup ($J_g$) and the bubble surface area flux. These researchers suggest that the gas holdup can be replaced by the bubble surface area flux to scale up the flotation cells, taking into account that the holdup measurements are easier to obtain.

### 3.2. Flotation studies at laboratory scale

The effect that the variation of the superficial velocity of the wash water ($J_{w1}$) between 0.25 cm s\(^{-1}\) and 0.4 cm s\(^{-1}\) (Fig. 3) had on (a) the metallurgical recovery of apatite, and contents of (b) P\(_2\)O\(_5\), (c) Fe\(_2\)O\(_3\), and (d) SiO\(_2\) was evaluated in the CCC.

The increase in $J_{w1}$ (for the range tested) causes a small variation in the metallurgical recovery of apatite – it fluctuates between 74.0% and 74.6%. The level of P\(_2\)O\(_5\) did not show a definite trend, ranging between 32.7% and 35% – the lowest level was observed for $J_w$ of 0.31 cm s\(^{-1}\). For the impurity levels of Fe\(_2\)O\(_3\) and SiO\(_2\), a clear pattern was seen of decreases in the levels of these penalizing elements corresponding to an increase in the $J_w$ value. The level of Fe\(_2\)O\(_3\) decreased from 8.2% ($J_w = 0.25$ cm s\(^{-1}\)) to 6.2% ($J_w = 0.40$ cm s\(^{-1}\)). Similarly, the level of SiO\(_2\) decreased from 3.5% to 2.7% (Fig. 3(c) and (d)).

In the 3PC flotation, evaluation was conducted based on the effect of the superficial velocity of wash water I ($J_{w1}$) added in the froth layer and by varying the superficial velocity of wash water II ($J_{w2}$) while keeping the other operating variables constant. $J_{w1}$ superficial velocities of 0.25, 0.31, and 0.40 cm s\(^{-1}\) (the same levels of $J_{w1}$ adopted for CCC studies), and $J_{w2}$ values of 0.0, 0.27, 0.31, and 0.40 cm s\(^{-1}\) were used.

Figs. 4.1–4.3 summarizes results obtained with the 3PC showing the effect of varying $J_{w1}$ and $J_{w2}$ on metallurgical recovery of apatite in concentrates and in the third product (drop-back, respectively).

For $J_{w1} = 0.31$ and 0.40 cm s\(^{-1}\), P\(_2\)O\(_5\) recoveries decreased somewhat with an increase in $J_{w2}$. For $J_{w1} = 0.40$ cm s\(^{-1}\), stabilization of values were found at $J_{w2}$ higher than 0.27 cm s\(^{-1}\). The maximum recovery achieved was approximately 73% P\(_2\)O\(_5\) and the minimum value was 55%.

In the drop-back flow, when $J_{w2} = 0.0$ cm s\(^{-1}\) was employed, P\(_2\)O\(_5\) grades reached about 18% and apatite recoveries ranged from 27% to 47% for the $J_{w1}$ (39% being the highest). Yet, metallurgical recoveries of apatite – about 45% – was obtained. The third product recovered (39% being the highest). Yet, metallurgical recoveries of apatite varied from 27% to 47% for the $J_{w2}$ range tested fairly lower than that obtained in the CCC (Fig. 3); higher values were obtained for $J_{w1} = 0.31$ cm s\(^{-1}\) and $J_{w2} = 0.40$ cm s\(^{-1}\).

#### 3.3. Flotation studies at pilot scale

Fig. 5 shows the effect of $J_{w2}$ on separation parameters (metallurgical recovery and grades) of phosphate in the 3PC cell. In the CCC, the P\(_2\)O\(_5\) concentrate grades varied between 35.5% and 36.5%; whereas in the 3PC values were always higher than 37% (39% being the highest). Yet, metallurgical recoveries of apatite varied from 27% to 47% for the $J_{w2}$ range tested fairly lower than that obtained in the CCC (65–75% R apatite).

In the case of $J_{w2} = 0.0$ cm s\(^{-1}\), a rich flotation concentrate content (39% P\(_2\)O\(_5\)) was obtained, whereas a metallurgical recovery of apatite – about 45% – was obtained. The third product recovered around 33% of the apatite with 18% P\(_2\)O\(_5\), higher than that of the feed which showed 11.4%, suggesting that this flow might be recycled in the flotation feed. In addition, Fe\(_2\)O\(_3\) grade was 27.4% compared to 8.3% in the feed; and the SiO\(_2\) content analyzed 12.7% against 13.7% in the feed. This alternative may reduce the low recoveries of apatite in this cell.

Table 4 shows data related to the selective character of the 3PC when enrichment ratios are compared with those obtained in the CCC.

Results shows that the P\(_2\)O\(_5\) enrichment ratios are always high for the 3PC (>3.28) and that the contents impurities are lower (lower enriching ratios). This is considered a very important in the fertilizing industry whereby high iron and silica contents may lead to costly penalties.
More, this table shows clearly the effect of the secondary wash water \( JW_2 \) on the 3PC performance. This secondary wash water reduces the degree of entrainment and entrapment of gangue slimes and impurities and is responsible for the bias water in the column. In this work, the higher the \( JW_2 \) the less was the hydraulic entrainment, and the cleaner were the concentrates. If this is not the case, and slimes are not present, this \( JW_2 \) parameter may be equal zero.

**Fig. 3.** Studies of phosphate flotation in the CCC, at bench scale: effect of the superficial velocity of the wash water \( JW_1 \) on recovery of apatite (a); on concentrate grades: \( P_2O_5 \) (b); \( Fe_2O_3 \) (c); and \( SiO_2 \) (d). Conditions: 40% solids (by weight), at conditioning stage and 15% at flotation; froth height = 18 cm; \( pH = 12; \) gelatinized corn starch = 1200 g t\(^{-1}\); soybean oil soap = 60 g t\(^{-1}\); \( J_a = 0.66 \text{ cm s}^{-1}; J_g = 0.49 \text{ cm s}^{-1}. \)

**Fig. 4.1.** Studies of phosphate flotation in the 3PC at bench scale, with \( JW_1 = 0.25 \text{ cm s}^{-1}. \) Effect of the superficial velocity of wash water \( JW_2 \) on: recovery of apatite (a); grades of concentrates and drop back products: \( P_2O_5 \) (b); \( Fe_2O_3 \) (c); and \( SiO_2 \) (d). Conditions: % solids: 40% at conditioning and 15% at flotation; Froth height = 18 cm; \( pH = 12; \) gelatinized corn starch = 1200 g t\(^{-1}\); soybean oil soap = 60 g t\(^{-1}\); \( J_a = 0.66 \text{ cm s}^{-1}. \)
The optimal superficial values (feed and air) were found (results not shown) to be as expected for this type of column (Rubio, 1996), where residence time and turbulence are meaningful factors. It is also believed that the so-called intermediate zone and its length are important in the 3PC column (Rubio, 1996, Valderrama and Rubio, 2008; Valderrama et al., 2011). If $J_{W2}$ is positive, this region becomes diluted compared with the collection zone, enabling bubble-hydrophobic particles to float faster.

Fig. 4.2. Studies of phosphate flotation in the 3PC at bench scale with $J_{W1} = 0.31$ cm s$^{-1}$. Effect of the superficial velocity of wash water $J_{W2}$ on: recovery of apatite (a); grades for concentrates and drop back products: $P_2O_5$% (b); $Fe_{2}O_3$% (c); and $SiO_2$% (d). Conditions: % solids: 40% at conditioning and 15% at flotation; froth height = 18 cm; pH = 12; gelatinized corn starch = 1200 g t$^{-1}$; soybean oil soap = 60 g t$^{-1}$; $J_a$ = 0.66 cm s$^{-1}$.

Fig. 4.3. Studies of phosphate flotation in the 3PC at bench scale with $J_{W1} = 0.40$ cm s$^{-1}$. Effect of the superficial velocity of wash water $J_{W2}$ on: recovery of apatite (a); grades for concentrates and drop back products: $P_2O_5$% (b); $Fe_{2}O_3$% (c); and $SiO_2$% (d). Conditions: % solids: 40% at conditioning and 15% at flotation; froth height = 18 cm; pH = 12; gelatinized corn starch = 1200 g t$^{-1}$; soybean oil soap = 60 g t$^{-1}$; $J_a$ = 0.66 cm s$^{-1}$. 

The optimal superficial values (feed and air) were found (results not shown) to be as expected for this type of column (Rubio, 1996), where residence time and turbulence are meaningful factors. It is also believed that the so-called intermediate zone and its length are important in the 3PC column (Rubio, 1996, Valderrama and Rubio, 2008; Valderrama et al., 2011). If $J_{W2}$ is positive, this region becomes diluted compared with the collection zone, enabling bubble-hydrophobic particles to float faster.
and low impurities contents in the phospha- 
and SiO2 (b):; grades for concentrates and drop back products: P2O5 (%); Fe2O3 (%); and SiO2 (%). Conditions: % solids: 40% at conditioning and 25% at flotation; froth height = 70 cm; pH = 12; gelatinized corn starch = 1400 g t−1; soybean oil soap = 70 g t−1; Jw1 = 0.65 cm s−1; Jw2 = 0.32 cm s−1.

Fig. 5. Studies of phosphate flotation in the 3PC at pilot scale, with a Jw2 = 0.37 cm s−1. Effect of the superficial velocity of wash water Jw2 on: recovery of apatite (a); grades for concentrates and drop back products: P2O5 % (b); Fe2O3 % (c); and SiO2 % (d). Conditions: % solids: 40% at conditioning and 25% at flotation; froth height = 70 cm; pH = 12; gelatinized corn starch = 1400 g t−1; soybean oil soap = 70 g t−1; Jw1 = 0.65 cm s−1; Jw2 = 0.32 cm s−1.

Table 4
Flotation of phosphate with the 3PC and CCC. Enrichment ratios (grade concentrate)/grade feed), at pilot scale.

<table>
<thead>
<tr>
<th>Jw2 (cm s−1)</th>
<th>3PC (Jw2: 0.37 cm s−1)</th>
<th>Enrichment ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>P2O5</td>
<td>SiO2</td>
</tr>
<tr>
<td>0.00</td>
<td>3.42</td>
<td>0.11</td>
</tr>
<tr>
<td>0.12</td>
<td>3.32</td>
<td>0.10</td>
</tr>
<tr>
<td>0.20</td>
<td>3.37</td>
<td>0.09</td>
</tr>
<tr>
<td>0.27</td>
<td>3.28</td>
<td>0.06</td>
</tr>
<tr>
<td>CCC (Jw2: 0.37 cm s−1)</td>
<td>3.21</td>
<td>0.12</td>
</tr>
<tr>
<td></td>
<td>2.81</td>
<td>0.10</td>
</tr>
<tr>
<td></td>
<td>2.72</td>
<td>0.12</td>
</tr>
<tr>
<td></td>
<td>2.67</td>
<td>0.13</td>
</tr>
</tbody>
</table>

4. Conclusions

The holdup values increased with DF 250 frother concentration and with the gas superficial velocity, and a linear relationship between these two gas dispersion values was found. The metallurgical recovery of apatite and the concentrate grades of P2O5 and impurities in the CCC varied little with enhancing the superficial velocity of the wash water, but in the 3PC, the contents of Fe2O3 and SiO2 lowered significantly with the extra washing water (Jw2). High concentrate grades of P2O5 (approximately 39%) and lower concentrations of impurities were always obtained in the 3PC at a lower metallurgical recovery, when compared with CCC. Results appear to indicate that the 3PC has higher potential in rougher-flash or cleaner flotation circuits, than other flotation cells, mainly because of the high P2O5 and low impurities contents in the concentrates attained meeting the strict requirements of the fertilizing industry. Also, recycling the third product (approximately 20% by weight) to flotation feed might enhance overall recovery.

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