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Intensification of humic acid extraction by pulse flow of vermicompost and sapropel slurries

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ABSTRACT

The extraction by pulse flow of slurry can be intensified in rotor–stator devices (RSDs). The humic acid (HA) yield after treatment with aqueous alkaline vermicompost slurry (VS) and sapropel slurry (SS) in the RSD is higher than that in the extraction apparatus with a helical ribbon impeller (HRI) at the same specific energy dissipation rate. The increase in the HA yield is caused by the destruction of particles and particle agglomerates of vermicompost and sapropel, deep penetration of the extractant in the pores of particles due to macro- and micropulsations in the fluid flow and cavitation.

1. Introduction

Humic acid (HA) is used in dyes, corrosion inhibitors, medicinal products, drilling fluids, plant growth stimulants (Tan, 2014). In industrial production HA is extracted from raw materials containing humic substances, using aqueous alkaline solution and vessels with anchor, gate or helical ribbon impellers. Helical ribbon impeller (HRI) can also be used in turbulent applications (Paul et al., 2004). HRI is effective for treatment of low-viscosity slurries, forming intensive radial and axis vortices in the vessel, and producing a mechanical action on the particles in the slurry by transverse blades and a helical surface of the blades. Particle size degradation, hydrodynamic flow pulsations, and cavitation increase the yield of water soluble HA due to an increase in the surface of contact between the phases, deep penetration of the solvent into the particle pores. These effects make it possible to obtain aqueous solutions of HA without using chemicals.

A promising direction is the development of devices with a multifactorial effect on slurry. The RSD operation principle is based on unsteady energy and substance flows (Paul et al., 2004; Bałdyga et al., 2007). RSDs have mechanical, hydrodynamic and acoustic effects on extraction processes in heterogeneous liquids (Promtov, 1997, 2009). To intensify the HA extraction process it is necessary to study RSD integrated effects on slurry.

In order to intensify solid-to-liquid extraction of a target component it is necessary to ensure that liquid penetrates into the pores of a solid body and is removed from the particle surface. In the RSD a heterogeneous liquid is subject to mechanical, hydrodynamic and acoustic effects through pulse, accelerating–decelerating nature of fluid flow motion in the device channels. Intensification of the extraction process in the RSD is determined by the energy expended on the slurry treatment.

The HRI is typically used when the viscosity of liquid is high. The viscosity of the VS and the SS was low. The HRI

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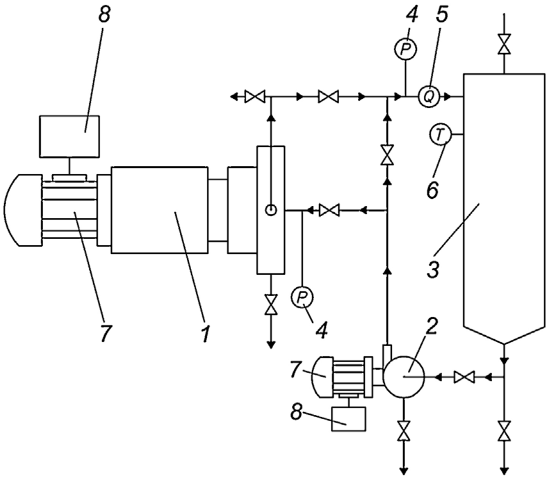

Fig. 1 – Schematic of the set-up for slurry treatment. 1 – RSD; 2 – pump; 3 – vessel; 4 – manometer; 5 – flowmeter; 6 – thermometer; 7 – electric motor; 8 – inverter.

was chosen because it had a strong mechanical effect on the suspension and agitated most of the fluid batch through the physical contact at a high rotational speed.

The aim of this work is to study the determinants of the HA yield from VS and SS for the RSD and in the HRI at the same level of energy consumption.

2. Experimental

The HA extraction from VS and SS was carried out in the prototype unit consisting of the industrial RSD (Fig. 1) and in the vessel with a HRI. The RSD has a cylindrical rotor and a cylindrical stator. The rotor and stator have rectangular slotted holes. The intensity of treatment in the RSD and in the HRI can be determined by the specific energy (dissipation energy). The specific energy in the RSD and in the HRI can be determined by the power spent by HRI or RSD and pump, W/kg. The power was measured by the inverter. The inverter displayed the total energy consumption, taking into account mechanical and electrical losses.

In the RSD we treated 90 kg of VS suspended in the solution with or without adding 1% alkali, and 90 kg of SS suspended in the solution with adding 1% alkali for 60 s at a temperature of 20 °C ± 2 °C. In the vessel with a HRI we treated 2 kg of VS in the solution with or without adding 1% alkali and 2 kg of SS in the solution with adding 1% alkali for 60 s at a temperature of 20 °C ± 2 °C. The ratio of components of the VS was 80% water or aqueous alkali solution and 20% dry vermicompost. The ratio of the components of the SS was 50% water or aqueous alkali solution and 50% sapropel gel.

The pressure was measured by testing manometers, and the temperature was measured by the SH-04016 digital single-channel temperature gauge with a relative error of measurement ±0.3%. The flow was measured by the VMG-50 flowmeter with a relative error of measurement ±2.0%. The frequency of rotor spinning and power of RSD was modified and measured by the VESPER EI-7011-100H inverter with a relative error of measurement ±0.01%. The inverter was connected via the RS485 communication protocol to the computer to control and record the motor shaft rotational speed and power consumption when operating the machine. The pump shaft speed was changed and measured with the TOSHIBA TOSVERT VF-S11 frequency inverter with a relative error of measurement ±0.01%. The centrifugal pump supplied the slurry through the RSD at a rate of $Q = 43 \pm 1\,\mathrm{m^3/h}$. The pressure at the inlet of the RSD was $P_1 = 0.45 \pm 0.02$ MPa, and the pressure at the outlet of the RSD was $P_2 = 0.05 \pm 0.01$ MPa.

The RSD parameters were as follows: the rotor diameter $d_R = 0.25$ m; the width $a = 0.003$ m and the height $h_c = 0.04$ m the rectangular channels of the rotor and stator; the rotor height $h = 0.05$ m; the gap between the rotor and stator $\delta = 0.0001$ m; the number of channels in the rotor and the stator $z = 36$; the length of the channel of the stator $l_S = 0.035$ m; the length of the channel of the rotor $l_R = 0.015$ m.

The slurries were treated using the universal laboratory AID type MPW-309 with the HRI for the HA extraction from VS and SS. The frequency of spinning and power of the HRI was changed and measured by the TOSHIBA VFnC3S inverter with a relative error of measurement ±0.01%.

The HRI parameters were as follows: the HRI diameter $d_S = 0.08$ m; the blade height $h = 0.18$ m; the number of blades $f = 2$; the blade pitch (height of one turn around the helix) $p = 0.05$ m; the blade-wall clearance $e = 0.015$ m; the blade width $w = 0.01$ m; the vessel height $H = 0.3$ m, the vessel diameter $D = 0.11$ m. The density and viscosity of the VS were $\rho = 1150\,\mathrm{kg/m^3}$; $\mu = 1.23 \cdot 10^{-3}$ Pa s. The density and viscosity of the SS were $\rho = 1050\,\mathrm{kg/m^3}$; $\mu = 1.17 \cdot 10^{-3}$ Pa s.

The fractional composition of fine particles of vermicompost was measured with the Micro Sizer 201C laser particle analyzer.

The concentration of the humic acid in the aqueous solution of VS or SS was calculated in compliance with ISO 5375-85 “Solid fuel. Method for determination of humic acids yield”.

3. Results and discussion

3.1. *Calculation of the specific energy of RSD and HRI*

The specific energy dissipated in the volume of the slurry is composed of the energy N_1 spent on the rotor spinning of the RSD, and energy N_2 expended by the external pump on pumping the slurry through the RSD. The specific energy dissipated in the volume of the slurry is expended on mechanical, hydrodynamic and acoustic effects.

The RSD specific energy dissipation rate ε_1, W/kg is:

$$\varepsilon_1 = (N_1 + N_2)/m_1, \tag{1}$$

where m_1 is the mass of treated slurry, kg; N_1 is the power required to spin the rotor, W; N_2 is the power expended by the pump on the slurry supply, W.

The power required to spin the rotor was determined by the formula (Promtov et al., 2015):

$$N_1 = Re^B \left(\frac{\delta}{R}\right)^{k_1} \left(\frac{az}{R}\right)^{k_2} \left(\frac{h}{R}\right)^{k_3} \omega^3 \rho R^4 h, \tag{2}$$

where $Re = \omega R^2 \rho/\mu$; ω is angular velocity of the rotor, $\mathrm{s^{-1}}$; R is the radius of the rotor, m; $B = -0.21$; $k_1 = 0.2$; $k_2 = 0.7$; $k_3 = -1.3$ are empirical coefficients.

The power N_2 expended by the pump on the slurry supply is calculated as

$$N_2 = \Delta P \cdot Q, \tag{3}$$

where Q is the flow rate, $\mathrm{m^3/s}$; ΔP is the pressure drop, Pa.

The pressure drop of RSD was measured directly:

$$\Delta P = P_1 - P_2, \tag{4}$$

where P_1 is the pressure at the inlet of the RSD, and P_2 is the outlet pressure of the RSD.

The HRI specific energy dissipation rate ε_2, W/kg is:

$$\varepsilon_2 = N_s/m_2, \tag{5}$$

where N_s is the power required to spin the HRI, W; m_2 is the mass of the slurry in the vessel, kg.

The HRI power calculation was performed according to (Paul et al., 2004; Tsui and Hu, 2011). The power consumed by the HRI was calculated as follows:

$$N_s = K_N \cdot \rho \cdot n^3 \cdot d_s^5, \tag{6}$$

where K_N is the power number; n is the rotational speed of the stirrer shaft, rps; d_s is the HRI diameter, m.

For fully turbulent hydrodynamic mode of HRI the calculation of K_N is difficult. For laboratory HRI ($Re = 10^4$–10^5) the experimental values of power consumption N_s satisfactorily agree with the calculated values with $K_N = 30$.

The rate of specific energy dissipation is the amount of power expended on treatment of m (kg) of the slurry in the RSD set-up or in the vessel with the HRI per unit of time. The treatment time in the RSD set-up and in the vessel with the HRI was the same.

Fig. 2 shows the dependency graphs of energy dissipation in the parameter n for VS and SS treatment in the RSD and the vessel with the HRI. The experimental values of the power expended by the RSD set-up on the slurry treatment were higher than the calculated ones, since the calculation did not take into account mechanical and electrical energy losses. The mechanical and electrical energy losses were $(0.15/0.2)(N_1 + N_2)$.

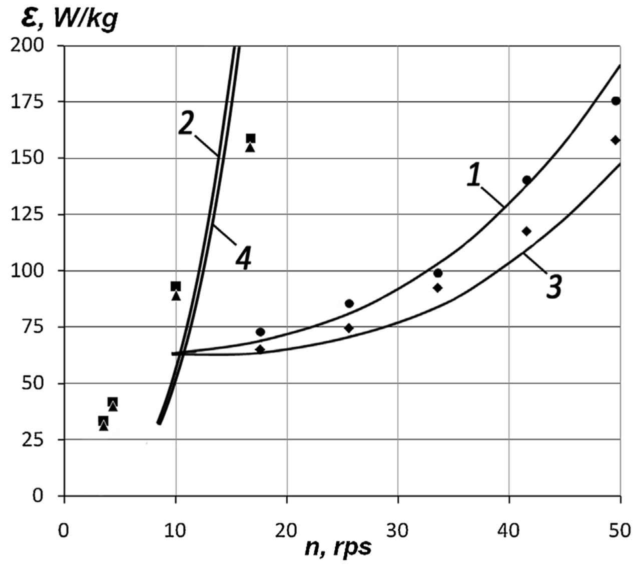

Fig. 2 – Dependency graphs of specific energy on rps: 1 – treatment of VS in RSD, experimental points – ●; 2 – treatment of VS in HRI, experimental points – ■; 3 – treatment of SS in RSD, experimental points – ♦; 4 – treatment of SS in HRI, experimental points – ▲.

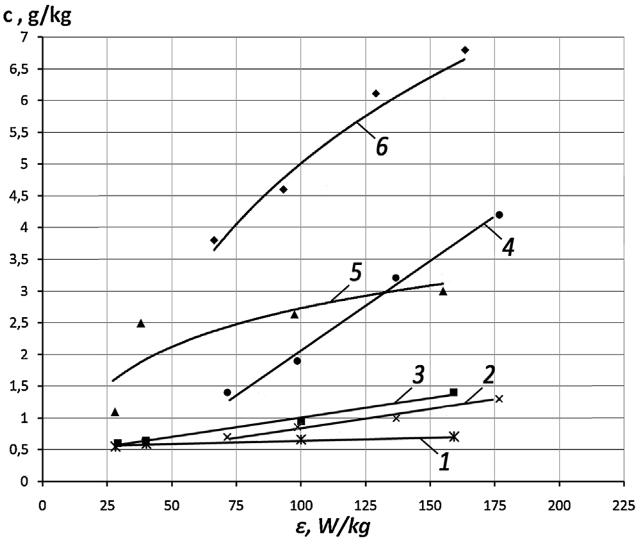

Fig. 3 – Dependency graphs of the HA concentration in the slurry on specific energy. Slurry treatment: 1 – VS in HRI (pH 7.5), experimental points – ✳; 2 – VS in RSD (pH 7.5), experimental points – ×; 3 – VS in HRI (pH 12.5), experimental points – ■; 4 – VS in RSD (pH 12.5), experimental points – ●; 5 – SS in HRI (pH 10.5), experimental points – ▲; 6 – SS in RSD (pH 10.5), experimental points – ♦.

3.2. *The concentration of HA in the slurry*

The experiments resulted in the HA extraction from VS and SS in the RSD and in the vessel with HRI; the data on the HA yield are presented in Fig. 3.

The HA yield for VS treatment in the RSD compared to that for VS treatment in the vessel with the HRI under the same level of specific energy was higher owing to the increase in the phase contact surface and vermicompost particle disintegration. Another factor that contributed to the intensification of the extraction process was intensive treatment and hydrodynamic cavitation effects.

The distribution curves of the fractional composition of vermicompost particles before and after treatment in the vessel with the HRI and RSD are shown in Fig. 4.

Disintegration of the slurry particles in the RSD is caused by mechanical action on the slurry particles. As the rotor turns,

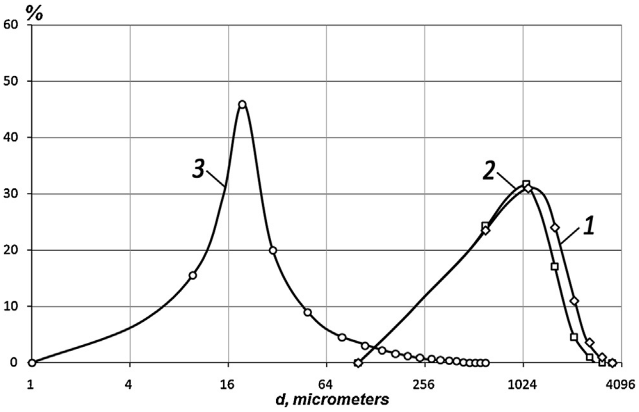

Fig. 4 – The distribution curves of the fractional composition of vermicompost particles (*d* is average particle size): 1 – before treatment; 2 – after treatment in the vessel with the HRI; 3 – after treatment in the RSD.

Table 1 – The Reynolds number, the cavitation number and the HA concentration in the slurry.

ε (W/kg)	70		115		160	
	RSD	HRI	RSD	HRI	RSD	HRI
Re (VS)	49×10^4	50×10^3	73×10^4	75×10^3	97×10^4	99×10^3
Re (SS)	47×10^4	48×10^3	70×10^4	72×10^3	93×10^4	95×10^3
C	0.86	40	0.82	18	0.78	10
c (g/kg) (VS)	0.65	0.6	0.9	0.68	1.23	0.7
c (g/kg) (VS + alkali)	1.4	0.82	2.48	1.1	3.75	1.4
c (g/kg) (SS)	3.82	2.42	5.48	2.85	6.58	3.1

high shear force and shear load occur in the gap between the rotor and stator. The working surfaces of the rotor and the stator have the impact force on the particles due to the mechanical contact. Cumulative streams occurring under collapse of cavitation bubbles have beating action on the particles and particle agglomerates. When processing VS in the vessel with the HRI, particles disintegration hardly occurred since the slurry was treated for 60 s at a low speed (1000 rpm) of the stirrer.

The intensity of cavitation in the hydrodynamic equipment was determined by the cavitation number. The cavitation number of the flow in the RSD was calculated by the formula (Promtov, 1997):

$$C=\frac{(P_s-P_\upsilon)}{P_m}, \tag{7}$$

where P_m is the amplitude of the pressure pulse in the stator channel, Pa; P_s is the static pressure in the stator channel, Pa; P_υ is the saturated vapor pressure, Pa. If $C\geq1$, cavitation in the fluid flow is not developed; if $C<1$, there is cavitation in the fluid flow, if $C\ll1$, cavitation in the fluid flow is developed.

The pulsed pressure generated in the stator channel was calculated by the formula (Promtov, 2009; Promtov et al., 2015):

$$P_m(t)=\rho\frac{dV}{dt}\left[\frac{S}{2\pi}\right]^{0.5}, \tag{8}$$

where S is the cross-section of the stator channel; for a rectangular cross-section of the stator channel $S=a\cdot h_c$; V is the fluid velocity in the stator channel, m/s.

A mathematical model describing the fluid flow through the channels of the rotor and the stator was based on the Bernoulli equation written in the non-stationary form (Promtov, 2009; Promtov et al., 2015):

$$l\cdot\frac{dV}{dt}+\lambda(t)\cdot\frac{l\cdot V^2}{2\cdot d_e}+\xi(t)\cdot\frac{V^2}{2}+\frac{B(t)\cdot\upsilon\cdot V}{2\cdot d_e}=\frac{\Delta}{\rho}, \tag{9}$$

where $l=l_S+\delta+l_R$; l_S is the length of the channel of the stator, m; l_R is the length of the channel of the rotor, m; δ is the size of the gap between the rotor and the stator, m; $V(t)$ is the average flow velocity in the stator channel cross-section, m/s; $\lambda(t)$ is the flow friction characteristic; d_e is the equivalent diameter of the stator channel, m; $\xi(t)$ is the total coefficient of local hydraulic resistance; $B(t)$ is the hydraulic resistance coefficient taking into account the pressure loss linearly dependent on the flow rate; υ is the fluid kinematic viscosity coefficient, m/s^2; Δ is the total pressure drop, Pa.

The amplitude of the pulse pressure in the channel of the stator was determined using Eq. (10) solved by the Runge–Kutta numerical method.

The cavitation number of the flow in the vessel with the HRI was calculated by the formula:

$$C=\frac{2(P_s-P_\upsilon)}{\rho V_s^2}, \tag{10}$$

where P_s is the static pressure in the vessel, Pa; $V=\omega_s d_s/2$ is the calculated speed of the slurry flow around the blade, m, ω_s is the angular velocity of the blade, s^{-1}.

The specific energy interval for the vessel with the HRI and the RSD under the same level was $\varepsilon=70$–160 W/kg. The Reynolds number (*Re*), the cavitation number (C) and the HA concentration (c) in the slurry for $\varepsilon=70$–160 W/kg are presented in Table 1.

The maximum value of the Reynolds number was equal to $Re_S=n\cdot d_S^2\cdot\rho/\mu\approx10^5$ and $Re_R=n\cdot d_R^2\cdot\rho/\mu\approx10^6$ when VS was treated in the vessel with the HRI and the RSD, respectively; n was in rps. The hydrodynamic mode for the vessel with the HRI and the RSD was fully turbulent (Paul et al., 2004).

For the RSD the hydrodynamic mode was more intense under developed cavitation. These are two factors that contributed to the disruption of the boundary layer from the surface of the particles, increasing the mass transfer of the HA from the particle surface.

4. Conclusions

Treatment of VS in the RSD causes particle size reduction, which leads to increased interfacial surface, opening of pores in the solid particles of the slurry. The intensive turbulent treatment of the slurry, pressure pulsations, flow rates, and cavitation effects contribute to a deeper penetration of the extractant into the pores of the VS and SS particles. At the expense of greater performance of the RSD, the specific energy values are comparable with those obtained during treatment in the vessel with the HRI. A combination of mechanical, hydrodynamic, acoustic and chemical effects on VS and SS treated in the RSD produces a synergetic effect for the HA extraction intensification. The HA yield under treatment of VS and SS in the RSD is on average ~1.6 times higher without alkali and ~2,2 times higher with the addition of alkali, in comparison with the treatment in the vessel with the HRI at comparable values of the specific energy.

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