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Mechanical and Thermal Activation of Navbahor Bentonite and Sapropel And the Use of Their Composites for Lead Immobilization in Contaminated Soil

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Abstract

Bentonite from the Navbahor deposit (Uzbekistan) and sapropel were activated mechanically in a planetary ball mill (24 full factorial design: 200/500 rpm, 30/90 min, ball-to-powder ratio 10:1/30:1, dry/wet) and thermally in a muffle furnace (bentonite 200–500 °C, sapropel 100–250 °C, 30–75 min), and the activated materials were combined into composites for Pb immobilization in soil spiked with 1500 mg/kg Pb. Milling raised the specific surface area (SBET) of bentonite from 50.13 to 70.97–102.31 m2/g and that of sapropel from 15.04 to 20.28–44.10 m2/g; wet milling gave a higher SBET than dry milling in every pair. Heating raised SBET of bentonite to 89.72 m2/g with little gain above 400 °C at 60–75 min, and that of sapropel to 40.10 m2/g at 250 °C, while its mean pore diameter fell from 19.05 to 11.53 nm. In the soil, citric acid- and Trilon B-extractable Pb decreased with a higher bentonite share (1:4 to 4:1), a higher dose (10–50 g/kg) and longer incubation (1–30 days). The composite of thermally activated bentonite (400 °C, 60 min) with milled sapropel at 4:1 and 50 g/kg left 22.48 % (citric acid) and 27.12 % (Trilon B) of the total Pb extractable after 30 days, against 24.14 % and 28.55 % for commercial humic acid at the same dose. On day 1 the composites were less effective than the commercial humic sorbents.

Keywords: Bentonite, sapropel, mechanical activation, thermal activation, specific surface area, lead immobilization, soil remediation.

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1. Introduction

Lead does not degrade in soil, and excavation or washing of large contaminated areas is expensive. In situ immobilization with mineral and organic amendments converts mobile Pb into less soluble forms and leaves the

topsoil in place [1]. Its effect is usually judged by the decrease of the fraction extracted by mild reagents, which is taken as an estimate of the bioavailable metal [2].

Clay minerals bind heavy metals by ion exchange, surface complexation and precipitation, and smectite-rich bentonites are among the most studied low-cost sorbents [3]–[5]. Humic substances add carboxyl and phenolic groups that chelate metal ions. Sapropel, an organic-mineral lake sediment, contains humic acids, nutrients and clay, and is already used as a soil amendment [6]. Uzbekistan has large bentonite reserves, including the Navbahor deposit in the Navoi region, so a bentonite–sapropel composite made from local raw materials could be a cheap alternative to imported humic sorbents.

2. Related Work

High-energy milling breaks clay particles, delaminates layers and creates fresh surface, and with prolonged milling it amorphizes the structure [7], [8]. Heating removes adsorbed and interlayer water and changes the surface area and porosity of bentonite; at the dehydroxylation stage the layered structure collapses [9]. The N2-BET surface area of bentonites also depends on the interlayer cation composition [10]. Calcined bentonite has been prepared at 400–500°C for Cu removal in packed beds [11], and Navbahor bentonite has been studied as an adsorbent for industrial wastewater [12].

For soil, humic acid-modified montmorillonite immobilized Cd and Hg and kept its effect under accelerated ageing [13]. Bentonite alone stabilized Cd and Pb in a co-contaminated paddy soil [14], a hydroxyapatite–bentonite–biochar mixture lowered available Cd and Pb in vegetable soil [15], and bentonite with biochar reduced Pb uptake and translocation in pea [16]. These studies used either a clay or an organic component, or commercial additives. Combined activation of a local bentonite and sapropel and its effect on Pb mobility in soil has not been reported.

3. Scope Of Research

The aim was to establish (i) how the mechanical and thermal activation regimes change the specific surface area and pore structure of Navbahor bentonite and sapropel, and (ii) how the bentonite:sapropel ratio, the dose and the incubation time of the activated composites affect the extractable fraction of Pb in contaminated soil, in comparison with three commercial humic sorbents.

4. Materials and Methodology

Alkaline-earth (Ca^{2+}/Mg^{2+}) bentonite was taken from the Navbahor deposit (Navoi region), air-dried below 40 °C for 3–4 days, ground in an agate mortar and sieved to <0.16 mm (sample B-0). Sapropel [sampling site to be confirmed by the authors] was dried at 105 °C for 24 h to <8 % moisture, ground and sieved to <0.25 mm (sample S-0). The bentonite is dominated by SiO_2 and Al_2O_3 (XRF, Fig. 1a), and the sapropel contains 32–40 % organic matter, including 12–18 % humic and 4–8 % fulvic acids (Fig. 1b).

Mechanical activation was carried out in a Retsch PM 400 planetary ball mill with steel balls of 10 mm diameter. Rotation speed (200 and 500 rpm), milling time (30 and 90 min), ball-to-powder ratio (BPR 10:1 and 30:1) and milling medium (dry, or wet with 10 % water) were varied at two levels, giving 16 regimes for each material. Wet-milled samples were dried at 50 °C. Thermal activation was carried out in a SNOL-7.2/1200 muffle furnace: 5–10 g of sample was heated at 12 °C/min to 200, 300, 400 or 500 °C (bentonite) or to 100, 150, 200 or 250 °C (sapropel), held for 30, 45, 60 or 75 min and cooled in the furnace. The upper limit for sapropel was set to protect humic acids, whose thermal decomposition starts at moderate temperatures [17].

Specific surface area, total pore volume and mean pore diameter were determined by N_2 adsorption at −196 °C (Micromeritics ASAP 2020; $P/P_0 = 0.01–0.99$; degassing at 150 °C for 4 h; BET method [18]). Each regime was run in three replicates and the mean is reported.

For the soil tests, composites were prepared from mechanically activated bentonite (500 rpm, 30 min, BPR 30:1, wet; denoted B-M) or thermally activated bentonite (400 °C, 60 min; B-T) and mechanically activated sapropel (500 rpm, 30 min, BPR 30:1, wet; S-M) at bentonite:sapropel mass ratios of 1:4 to 4:1. They were added at 10–50 g/kg to soil containing 1500 mg/kg Pb [soil type, Pb source and incubation moisture/temperature to be stated by the authors] and incubated for 1, 15 and 30 days. Calcium humate, oxidized coal and humic acid were tested at the same doses. Pb was extracted with 2 % citric acid and with 0.2 M Trilon B (Na_2EDTA) and is expressed as a percentage of the total Pb in the soil [determination method for Pb to be stated by the authors].

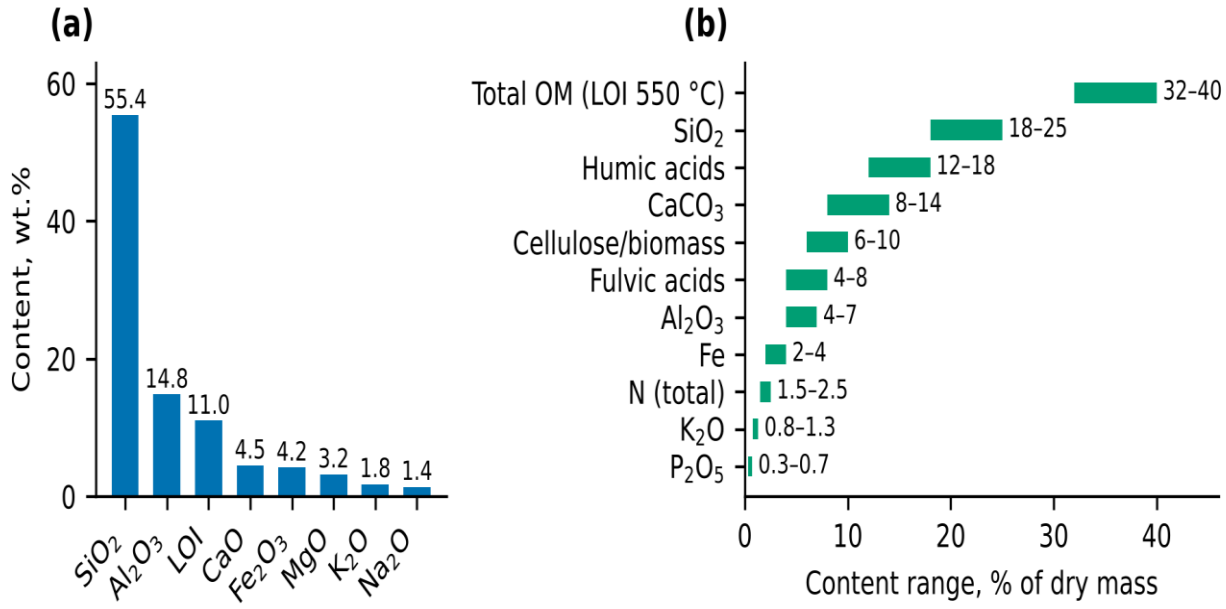


Fig. 1. Composition of the raw materials:

(a) Navbahor bentonite (XRF), LOI – loss on ignition;

(b) sapropel, ranges on a dry basis, OM – organic matter.

5. Experimental Results and Discussion

A. Raw materials. Natural bentonite had SBET = 50.13 m2/g, a pore volume of 0.081 cm3/g, a mean pore diameter of 5.01 nm and a basal spacing d001 = 1.55 nm. Sapropel had a much lower SBET of 15.04 m2/g and a pore volume of 0.040 cm3/g but larger pores (19.05 nm), because its gel-like organic matrix restricts N2 access to the mineral surface.

B. Mechanical activation. All milling regimes increased SBET (Fig. 2a, b). For bentonite it rose from 50.13 to 70.97 m2/g at the mildest regime (200 rpm, 30 min, BPR 10:1, dry) and to 102.31 m2/g at the most intensive one (500 rpm, 90 min, BPR 30:1, wet). Raising the speed from 200 to 500 rpm added 11.07–17.23 m2/g at otherwise equal settings. Wet milling gave a higher SBET than dry milling in all eight pairs, by 1.18–7.10 m2/g for bentonite and by 2.53–5.07 m2/g for sapropel. Water films between particles reduce agglomeration during milling, which explains this gain. For sapropel, SBET rose from 15.04 to 20.28 m2/g at the mildest regime and to 44.10 m2/g at the most intensive one.

For bentonite the pore volume did not follow SBET at the most intensive regime: at 500 rpm and 30 min with BPR 30:1 (wet) it reached 0.22 cm3/g at SBET = 99.38 m2/g, whereas after 90 min it was 0.17 cm3/g at 102.31 m2/g (Fig. 3a). The longer treatment therefore produced more surface but closed part of the pore space, which agrees with the agglomeration and amorphization reported for prolonged milling of clays [8]. The 500 rpm, 30 min, BPR 30:1, wet regime (B-M) was selected for bentonite, and the same regime (S-M; 42.07 m2/g, 0.123 cm3/g) for sapropel.

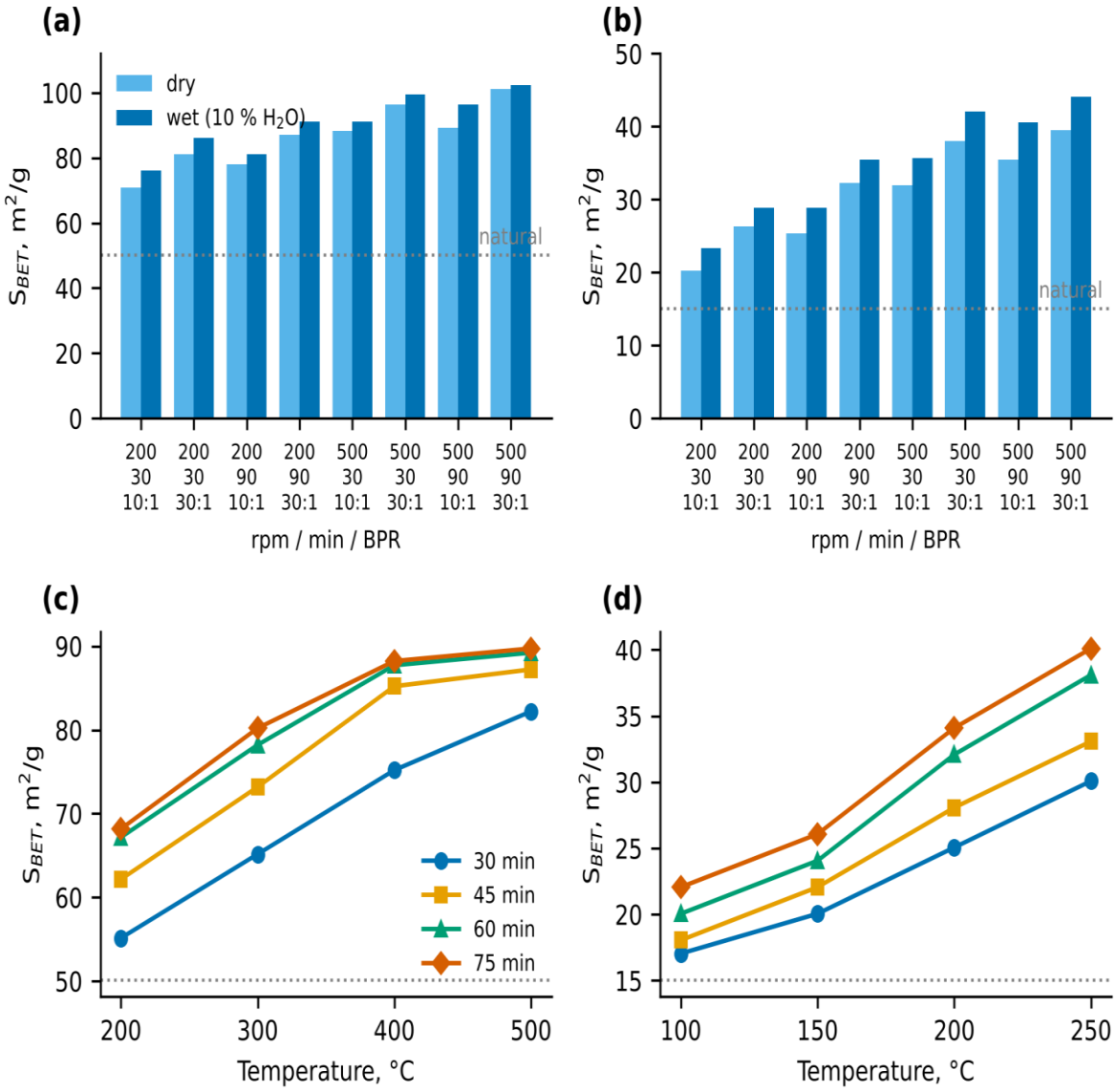


Fig. 2. Specific surface area after activation:

(a) bentonite and (b) sapropel milled under dry and wet conditions (rotation speed, rpm / time, min / BPR);

(c) bentonite and (d) sapropel heated at different temperatures and times. Dotted line – natural material.

C. Thermal activation. Heating increased SBET of bentonite with both temperature and time (Fig. 2c). At 30 min it rose from 55.14 m2/g at 200 °C to 82.21 m2/g at 500 °C. At 60 min the values were 67.17, 78.20, 87.72 and 89.22 m2/g at 200, 300, 400 and 500 °C, so only 1.50 m2/g was gained between 400 and 500 °C, and extending the time from 60 to 75 min at 500 °C added 0.50 m2/g. The pore volume behaved in the same way (0.157 cm3/g at 400 °C and 0.159 cm3/g at 500 °C, 60 min). This plateau corresponds to the removal of interlayer water before dehydroxylation [9], and 400 °C for 60 min (B-T) was chosen as the less energy-intensive regime.

For sapropel, SBET increased from 15.04 to 30.08 m2/g at 250 °C and 30 min and to 40.10 m2/g at 250 °C and 75 min (Fig. 2d). The mean pore diameter decreased at the same time from 19.05 nm to 13.03 nm (250 °C, 30 min) and 11.53 nm (250 °C, 75 min) (Fig. 3b). Heating thus opened smaller pores, probably by removing water and labile organic matter from the gel. Across all four series, pore volume rose with SBET (Fig. 3a; r = 0.88 for milled bentonite and 0.96–0.99 for the other three series).

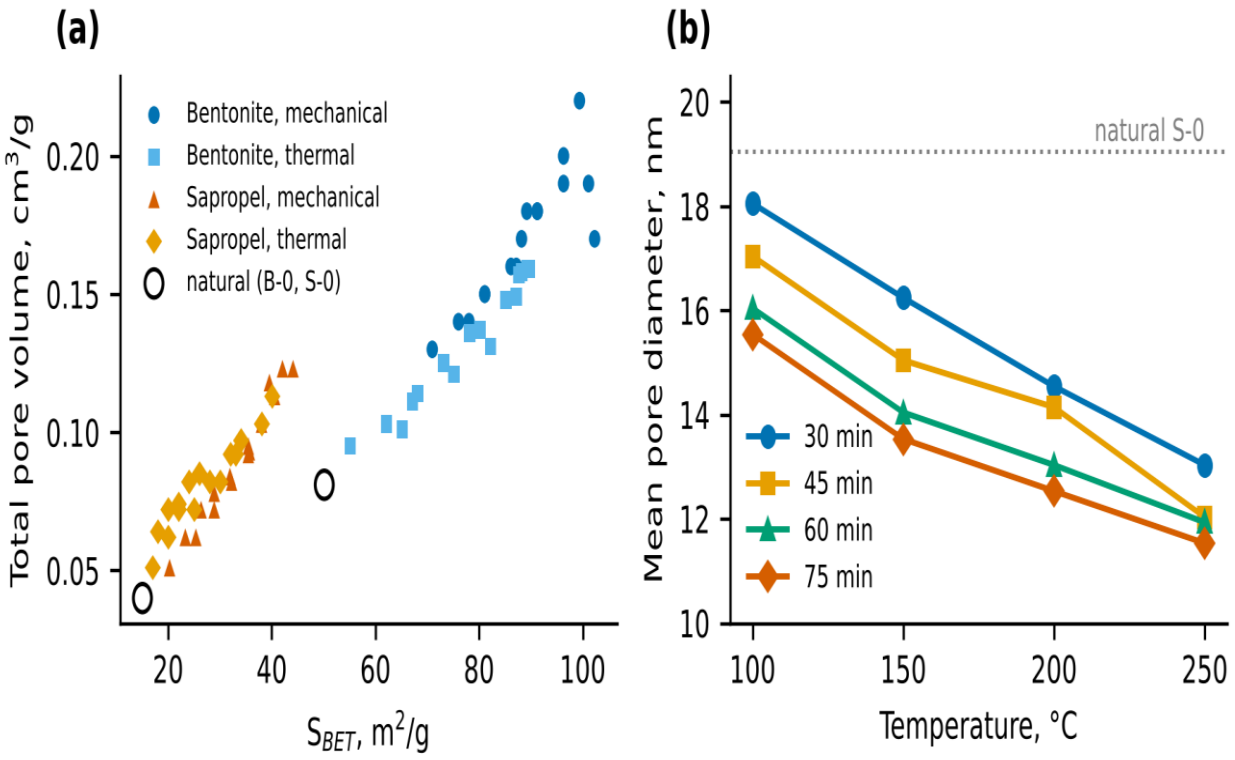


Fig. 3. Pore structure of the activated materials:

(a) total pore volume versus SBET for all mechanically and thermally activated samples;

(b) mean pore diameter of sapropel versus heating temperature and time.

D. Effect of ratio and dose on Pb extractability. After 30 days at 30 g/kg the extractable Pb decreased steadily as the bentonite share increased (Fig. 4a). With B-T/S-M, Trilon B-extractable Pb fell from 41.04 % at 1:4 to 30.71 % at 4:1 and citric acid-extractable Pb from 35.74 % to 25.40 %; with B-M/S-M the corresponding values were 44.59 → 33.36 % and 38.95 → 27.69 %. Citric acid extracted 4.5–6.3 percentage points less Pb than Trilon B in every ratio–dose combination after 30 days, as expected for a weaker complexing agent. The stronger effect of bentonite-rich mixtures points to retention on the montmorillonite layers by ion exchange and inner-sphere complexation [4], whereas sapropel contributes mainly organic matter and humic acids.

Increasing the dose from 10 to 50 g/kg at 4:1 lowered Trilon B-extractable Pb from 37.16 to 27.12 % (B-T/S-M) and from 40.36 to 29.46 % (B-M/S-M) (Fig. 4b). The successive 10 g/kg steps lowered it by 3.53, 2.92, 1.90 and 1.69 percentage points for B-T/S-M, so each additional portion of sorbent gave a smaller effect. In all 35 ratio–dose combinations after 30 days, the composite with thermally activated bentonite left 2.03–4.27 percentage points less extractable Pb than the composite with milled bentonite, although B-T had a lower SBET (87.72 m2/g) than B-M (99.38 m2/g). Surface area alone therefore does not determine Pb retention, and the better-preserved crystalline structure after moderate heating may be one reason; this needs confirmation by XRD of

the soil-incubated material.

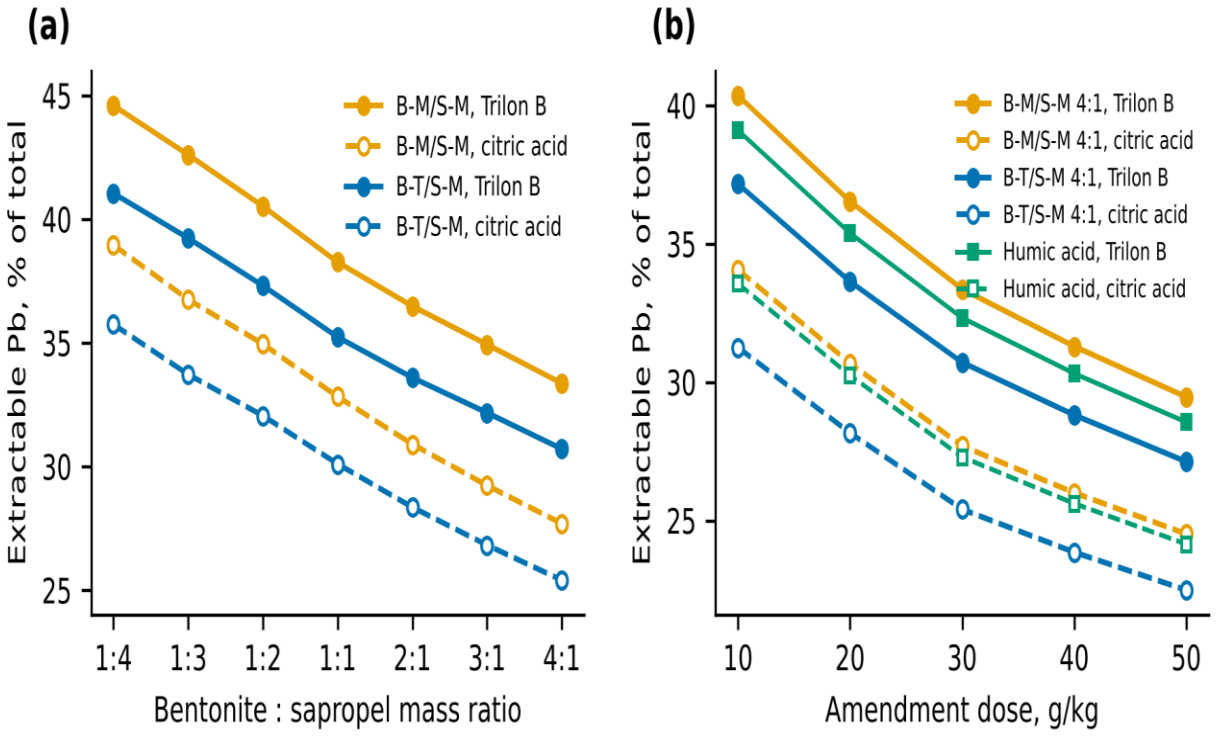


Fig. 4. Extractable Pb (percentage of total Pb, 1500 mg/kg) in soil after 30 days:

(a) as a function of the bentonite:sapropel ratio at 30 g/kg;

(b) as a function of the dose at 4:1, with commercial humic acid for comparison. Solid lines – 0.2 M Trilon B, dashed lines – 2 % citric acid.

E. Incubation time and comparison with commercial sorbents. The composites acted more slowly than the commercial humic sorbents (Fig. 5a). At 1:1 and 30 g/kg, Trilon B-extractable Pb with B-T/S-M was 57.64 % on day 1, 41.82 % on day 15 and 35.23 % on day 30, a decrease of 22.41 percentage points; with humic acid it was 41.27, 36.51 and 32.32 %, a decrease of 8.95 points. On day 1 at 10 g/kg, all three commercial sorbents left less extractable Pb than the 4:1 composites (49.93–55.45 % against 60.80–66.04 %), but by day 30 the order had changed: B-T/S-M (37.16 %) was below humic acid (39.10 %), oxidized coal (41.01 %) and calcium humate (43.43 %), while B-M/S-M (40.36 %) was between humic acid and oxidized coal (Fig. 5b). The slow stage is consistent with gradual diffusion of Pb into the clay structure, which is slower than complexation by dissolved humic matter.

At the highest dose and after 30 days (Fig. 5c), B-T/S-M at 4:1 gave the lowest values of all five sorbents: 22.48 % citric acid-extractable and 27.12 % Trilon B-extractable Pb, compared with 24.14 % and 28.55 % for humic acid, 25.64 % and 29.93 % for oxidized coal and 27.30 % and 31.70 % for calcium humate. B-M/S-M (24.51 % and 29.46 %) was close to humic acid. Expressed in concentration, 22.48 % corresponds to 337 mg/kg of citric acid-extractable Pb out of 1500 mg/kg. Results from other studies cannot be compared directly because extractants, soils and Pb levels differ, but bentonite and humic amendments in [13]–[16] were also effective at doses of a few percent, which is in line with the 30–50 g/kg range found here. The composites therefore need time to act and are suited to treatment ahead of the growing season rather than to rapid response.

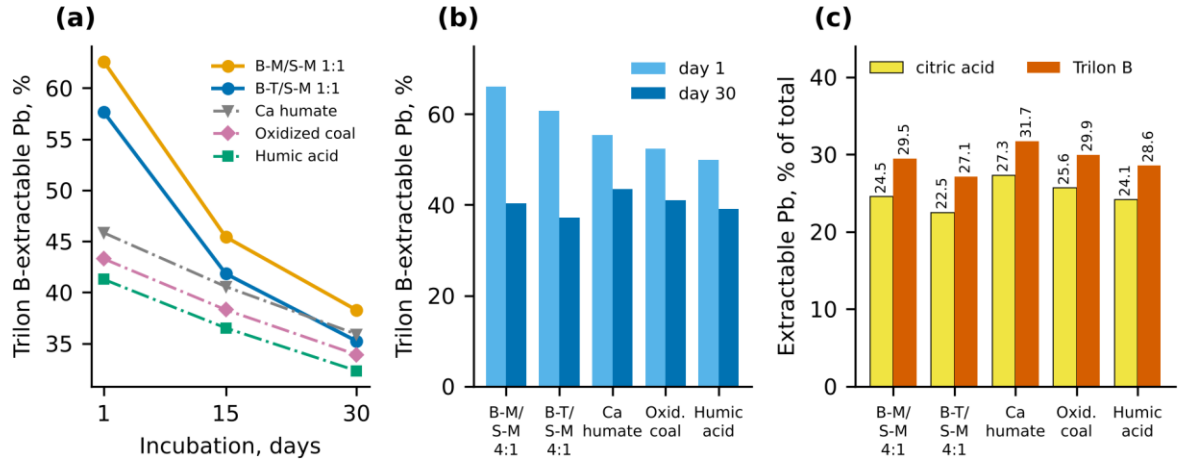


Fig. 5. Comparison of the composites with commercial humic sorbents:

(a) Trilon B-extractable Pb versus incubation time at 30 g/kg (composites at 1:1);

(b) Trilon B-extractable Pb on days 1 and 30 at 10 g/kg;

(c) citric acid- and Trilon B-extractable Pb after 30 days at 50 g/kg.

The study has limitations. Only one Pb-spiked soil and one contamination level were tested, the extractable fractions are given relative to total Pb without an unamended control, and the binding forms of Pb were not identified by sequential extraction or spectroscopy. These points should be addressed before field application.

6. Conclusion

Mechanical activation raised the specific surface area of Navbahor bentonite from 50.13 to 102.31 m2/g and of sapropel from 15.04 to 44.10 m2/g; wet milling was more effective than dry milling in every regime pair, and the 500 rpm, 30 min, BPR 30:1, wet regime gave the largest pore volume of bentonite (0.22 cm3/g). Thermal activation of bentonite reached a plateau at 400–500 °C and 60–75 min (87.72–89.72 m2/g), and heating of sapropel at up to 250 °C raised its surface area to 40.10 m2/g while reducing the mean pore diameter from 19.05 to 11.53 nm. In Pb-contaminated soil the extractable Pb decreased with the bentonite share, the dose and the incubation time. The composite of bentonite heated at 400 °C for 60 min with milled sapropel (4:1, 50 g/kg, 30 days) left 22.48 % of the total Pb extractable by citric acid and 27.12 % by Trilon B, lower than commercial humic acid, oxidized coal and calcium humate at the same dose, but it acted more slowly than these sorbents in the first days.

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