



# Recovery of phosphorus from incinerated sewage sludge ash by combined two-step extraction and selective precipitation

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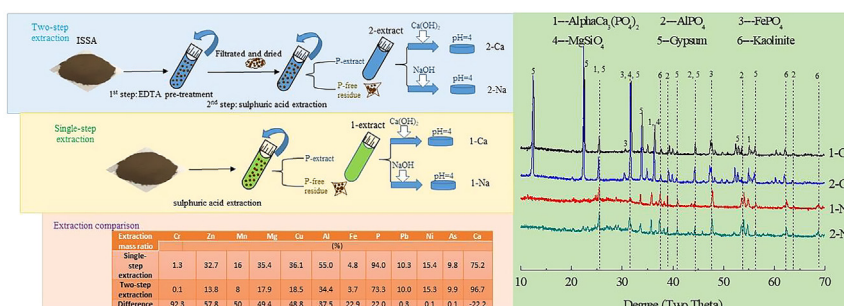
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## HIGHLIGHTS

- Extraction conditions of two-step extraction method were optimized.
- P-precipitates were obtained by adding  $\text{Ca}(\text{OH})_2$  and  $\text{NaOH}$  to P-extracts.
- Two-step extraction method can produce high purity of P leachate.
- Adding  $\text{Ca}(\text{OH})_2$  for pH adjustment can induce more Ca-P formation.
- Two-step P-precipitate by adding  $\text{Ca}(\text{OH})_2$  showed highest purity and contained effective soil restorer.

## GRAPHICAL ABSTRACT



## ARTICLE INFO

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## ABSTRACT

The recovery of phosphorus (P) from acid extracts of incinerated sewage sludge ash (ISSA) is invariably complicated by the co-dissolution of metal(loid)s. An extra purification treatment step is thus needed, which reduces the efficiency of P recovery. This study developed a two-step extraction method which can produce highly purified P-extract where ISSA is treated first with ethylenediaminetetraacetic acid (EDTA) and then with sulphuric acid. The recovery conditions of this two-step extraction method were optimized (pre-extraction by 0.02 mol/L of EDTA at a liquid to solid ratio (L/kg) of 20:1 during 120 min, then extraction by 0.2 mol/L sulphuric and at a liquid to solid ratio of 20:1 during 120 min). Compared with direct extraction by sulphuric acid (namely, single-step leaching), the two-step extraction method dissolved 2.34 mmol/kg ISSA less of P, but with 5.16 mmol/kg ISSA less of metal(loid) contamination.

Furthermore, the potential for co-precipitation of P and the metal(loid) contaminants in the extracts (both single-step extraction and the two-step extraction methods) was examined from pH 2 to 12 (adjusted by addition of  $\text{NaOH}$  and  $\text{Ca}(\text{OH})_2$ ). By applying  $\text{Ca}(\text{OH})_2$  and at the optimal pH of 4, the two-step extraction method could significantly increase Ca-P in the precipitate and notably decreased the metal(loid) contaminants by 50% compared to the single-step method.

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## 1. Introduction

The exhaustion of limited natural phosphate rock resources and the one-way flux of phosphate rock from highly concentrated ores to diffuse low level concentrations in the environment stimulate the motivation of recycling P from waste streams and convert a linear consumption of P resources into a partially circular consumption model. Current worldwide available phosphate ore reserves are predicted to be depleted within the next 50–100 years [1–3] while Chinese phosphate ore might be exhausted within 37 years [4]. After mining, only 10% of P is consumed by human activities [5], and then the consumed-P and waste-P are disposed as waste streams [6]. If properly collected, these P-wastes typically end up in the sewage of waste-water treatment plants, where the vast majority of P is removed in the sewage sludge [7]. Although land-spreading remains the dominant form of sewage sludge disposal, during the last 20 years there has been a trend at a global level away from landfilling and sea disposal towards incineration of sewage sludge [8]. Incineration is especially attractive in densely-populated regions and effectively reduces the volume of waste, eliminates odour problems, produces a sterile ash and allows for potential energy recovery from the organic solids in the sludge [9,10]. Almost all of the P in sewage sludge remains in the inorganic fly ash produced by sewage sludge incineration [3,11,12].

Incinerated sewage sludge ash (ISSA) consists of small glassy particles partially sintered together into irregularly shaped porous agglomerates containing Fe, Al, Si and P [13,14]. The P is preferentially deposited on the surface of particles, thus facilitating ease of recovery [5,10,15]. The main crystalline phases containing P in ISSA are whitlockite, beta-tricalcium phosphate mineral and  $\text{AlPO}_4$  [5,16,17]. However, the presence of Mg and Fe can also influence the type of P phases formed.

According to previous studies, either thermal treatment or wet-extraction is used to recover P from ISSA [10,18,19]. Thermal methods have limitations due to high energy consumption and unsatisfactory removal results for non-volatile metals (Ni, As, Mo, etc) [20]. Wet-extraction is more common and mineral acids have been shown to be the best recovery agents because they readily dissolve P but the co-dissolution of metal(loid)s is a problem [10]. However, alkaline solutions induce a low P solubility although the co-dissolution of metal(loid)s is also low [20–24]. The presence of metals such as Zn, Cd, Mg and Pb, reduce the quality of phosphate fertilizer [25]. Efforts to eliminate these metal(loid)s have included adsorption by organic and inorganic media (activated carbon, biochar, modified silicate, etc.) [26–29], cation-exchange resins [3], solvent extraction [10] and ion migration in electric fields [30].

Each purification method has specific limitations. For example, removal efficiencies by cation adsorption in a highly acidic solution are impeded by the high concentration of  $\text{H}^+$  ions. Organic solvent extraction is not sufficiently efficient and also requires an additional step for P extraction from the solvent and solvent recovery before a phosphoric acid product can be obtained [10,23]. The removal of metal ions by direct current was found to be limited by high energy consumption and low efficiency [31,32]. Hence, it is necessary to find new methods which can effectively extract P with high purity through a simple process route.

Our previous study has identified that ethylenediaminetetraacetic acid (EDTA) and sulphuric acid could effectively leach metal(loid)s and

P, respectively [14]. EDTA is applied mostly in soil washing and can remove metal(loid)s effectively [33–35], while sulphuric acid was found to be an optimal P extraction agent [14]. Consequently, it makes sense to combine the properties of these two extractants in a sequential procedure.

Obtaining a solution with low concentrations of metalloids and a high concentration of P is only part of the challenge. The next step is to convert that solution in a useful P-based product. Previous studies have demonstrated that P co-precipitates with Al, Fe and Ca at pH above 4 [36,37]. Of the three kinds of P-precipitation, only Ca-P is an effective constituent for fertilizer application and therefore significant efforts have been made to recover Ca-P directly or produce it from other P-phases. However, direct recovering of Ca-P has low efficiency while the complex transforming process would result in P loss [36,37].

Consequently, this study has assessed the feasibility and efficiency of generating a solid CaP-based product by pH adjustment (by addition of NaOH or  $\text{Ca}(\text{OH})_2$ ) of the less contaminated solution obtained from the two-step extraction procedure. The overall aim of the research is to develop a commercially viable process for extricating P from sewage sludge ash so that P can become part of a circular economy.

## 2. Materials and methods

The ISSA used in this study was from the Sludge Treatment Facility (T-park) in Hong Kong and it has been characterised in previous studies [14,15]. The  $\text{H}_2\text{SO}_4$  (AR, 98%) and EDTA (ACS, 99.5%) were supplied by RCI Labscan limited and Anaqur Chemicals, respectively.

### 2.1. Extraction process by two-step and single-step methods

The optimized conditions for metal(loid)s removal and P extraction using only EDTA or only sulphuric acid have been investigated in our previous study [14]. However, when considering these extractions in sequence, it is evident that the first step with EDTA influences the mobility of heavy metals in the solids that are fed to the second step [38,39]. It therefore becomes necessary to reoptimize the conditions for both steps, with the aim of achieving the highest P recovery with the lowest metal(loid) impurities.

The two-step extraction method is illustrated in Fig. 1. The variables of the EDTA (1st step) single-factor optimization conditions are shown in Table 1, which includes concentration, liquid to solid ratio and reaction time. For extraction experiments, 1.0 g of dried and homogenized ISSA was weighed ( $\pm 0.005$ ) and transferred into a 50 ml centrifuge tube. The EDTA reagent (concentration and volume according to Table 1) was added, and the tube placed on an end-over-end mechanical rotator at 30 rpm for continuous mixing (contact times are given in Table 1). After the EDTA extraction, the mixture was centrifuged at 4000 rpm for 10 min. The supernatant was withdrawn, filtered using a 0.45- $\mu\text{m}$  mixed cellulose ester membrane filter paper and analyzed for metal(loid)s by inductively coupled plasma optical emission spectroscopy (ICP-OES, SPECTROBLUE FMX 36) and for P by colorimetry after molybdenum blue complex formation. Prior to the second step, the solid residues were washed with deionized water to remove excess EDTA and then dried at 105 °C overnight.

The second step involved adding sulphuric acid under the optimized conditions (0.2 mol/L, 2 h, 20:1 of liquid to solid ratio) as identified in a previous research [14]. For comparison, a single step extraction was

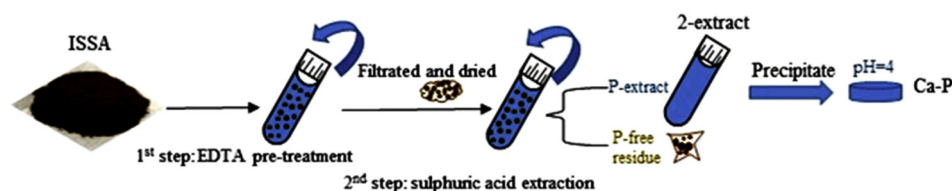


Fig. 1. Process of two-step extraction.

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