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A review of phosphorus recovery methods at various steps of wastewater treatment and sewage sludge management. The concept of “no solid waste generation” and analytical methods

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a b s t r a c t

Phosphorus deposits around the world are rapidly depleting, therefore phosphorus recovery methods are gaining more and more interest both in science and industry. This article presents the main methods of phosphorus recovery from sewage sludge. The described approaches are divided in two groups: phosphorus recovery from sewage sludge and leachate, and recovery of phosphorus from sewage sludge ashes. The latter seems to have more advantages connected with both ecological and economical aspects. The need for development of “no solid waste generation” strategy is becoming more and more urgent. The concept of comprehensive management of all solid residues after what is currently considered the most ecological process of sewage sludge incineration connected with phosphorus recovery based on acidic extraction, is described in the article. Solid residues after phosphorus recovery from sewage sludge ashes by means of acidic extraction can be stabilized with solid residues after sewage sludge incineration exhaust gas treatment. Such an approach may enable production of phosphoric raw material together with stabilized construction material. Advantages and disadvantages of the discussed approaches are given. An analysis of the composition of ashes produced in different sewage sludge treatment plants indicates that the proposed technology could be successfully applied in most of such units, especially because the concentrations of elements such as K, Mg, Na, P are sufficiently high, respectively 1.5e12.1 g/kg; 9.9e14.9 g/kg; 3.6e13,3 g/kg and 27,4e99,0 g/kg. However, a phosphorus recovery method should be developed separately for each treatment plant. Only then all comprehensive management methods will be ecologically and economically justified. Analytical methods which could be of use at every step of designing a proper phosphorus recovery process are described.

Keywords: Phosphorus recovery, Sewage sludge ash, Sewage sludge, Struvite, Heavy metals

Contents

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1. Introduction

Phosphorus is increasingly recognized as a strategic raw material. The main reason for this is the growing world population. It is estimated that in the middle of this century our population will exceed 9 billion, which means that food production will have to be increased by almost 30%. Currently, about 82% of the mined phosphorus is used in agriculture, while 7% is used for the production of animal feed. The remaining 11% of the mined phosphorus is used in industry and medicine for the production of pharmaceuticals, oils, detergents, or even textiles (Cordell et al., 2009; Sorensen et al., 2015; de-Bashan and Bashan, 2004). The demand for food increases with population growth, which is evident in the third world countries, whereas in the developed countries the importance of industry is quickly increasing, which causes phosphorus consumption to increase faster and faster. For this reason, according to the International Water Management Institute, by 2050 the production of phosphorus will grow by 70% overall and even by up to 100% in developing countries, in order to satisfy the rapidly growing demand for phosphorus (Guedes et al., 2014; Zhou et al., 2016). An equally important factor in determining the strategic nature of this element is the fact that natural deposits of phosphorus are not evenly distributed throughout the Earth. The main global phosphate rock deposits are in Morocco, the US, China, South Africa and Jordan, 74% of which are found in Morocco alone (Nakakubo et al., 2012; Cordell et al., 2009; Sorensen et al., 2015; Kataki et al., 2016). The largest amounts of phosphorus are mined in China, the United States has the largest reserves, while South Africa exports the largest quantity. Therefore, the geopolitical situation can have a significant impact on the availability and price of phosphorus, not only in Europe, but in every corner of the world (Guedes et al., 2014).

All over the world there are heated debates about when the global phosphorus resources will be exhausted. According to optimistic estimations of apatite ores, which are the main natural phosphorous deposits, it will not happen for at least 200 years (Tan and Lagerkvist, 2011). Many scientists claim, however, that the deposits will be exhausted as early as in 100–130 years (Li et al., 2014). It should be mentioned that these are reports from about a decade ago, and forecasts made after 2008 are increasingly less optimistic. Currently, there are even mentions that the natural deposits of phosphorus will suffice for only 50–60 years (Tao and Xia, 2007). Some literature data report that by 2050 only 18% of phosphorus deposits will be exhausted, others claim that by the year 2200 the quality of phosphorus ores will be unsatisfactory, making the 11 000 million tons of remaining global phosphorus ores impossible to exploit. The issue of when the deposits of this raw material will really deplete can still be a subject of discussion (Sorensen et al., 2015; Shu et al., 2006; Kataki et al., 2016). However, the fact is that the quality of the available deposits of phosphorus is getting lower. It is already known that only 20% of the phosphorus which is cheap to extract may be suitable for use in agriculture. The rest of the deposits contain too high levels of heavy metals, especially cadmium, and radioactive elements, such as uranium or radon (Pettersson et al., 2008; Cordell et al., 2009; Weigand et al., 2013).

Factors such as the quality of the natural deposits of phosphorus, their availability, and even the geopolitical situation, cause the prices of phosphorus fit for use in agriculture to grow fast. From 1950 to 2000 they increased tenfold; in 2007 alone they grew by about 200%, and in the years 2007–2008, in less than 14 months they rose by 700–800% (Tan and Lagerkvist, 2011; Guedes et al., 2014). That is why scientists are increasingly interested in the methods of phosphorus recovery. Unfortunately, there is still a lack of legal regulations concerning recovery of phosphorus from waste materials (Biplob et al., 2009; Sorensen et al., 2015; Pettersson et al., 2008; Cordell et al., 2011).

Today there are more than 30 kinds of technologies of phosphorus recovery from sewage sludge and new ones are constantly being created (Cordell et al., 2011). All of them carry both risks and opportunities. The biggest disadvantage of the previously presented works on phosphorus recovery methods is a lack of complex management of waste materials and residues which are inevitably produced during the processes. Moreover, some of them are economically unjustified or designed in a way that cannot be implemented on the technical scale. The concept proposed in this paper assumes holistic management of all streams of solid waste generated during the phosphorus recovery process and sewage sludge thermal utilization. Only with the introduction of individual solutions it is possible to design an ecological and economical method of phosphorus recovery connected with the management of the remaining sludge and other process waste generated during sewage treatment.

2. Methods and scope

This study focuses on describing methods of phosphorus recovery from sewage sludge and presenting the concept of "no waste generation". Advantages and disadvantages of the described approaches are presented together with thorough evaluation of selected technologies. Analytical methods are described as powerful tools supporting both phosphorus recovery and the whole sewage sludge management process. The review is based on literature from all over the world but focusing mainly on reports from Europe and Asia since scientists from those regions show high interest in the presented subject. Studies published in technical journals and books are also mentioned. Flow sheets and as-built documentations were used to describe the proposed concept of "no solid waste generation" technology. Additional data on the elemental composition of sewage sludge and ashes were obtained from two polish facilities which are interested in the proposed technology: Sewage Sludge Treatment Plants "Wschód" in Gdańsk and Group Sewage Sludge Treatment Plants in Łódź.

3. Phosphorus in sewage sludge

Many organic waste materials contain significant amounts of phosphorus in various forms: organic, poorly absorbed by plants, and inorganic, better assimilated by plant organisms. Sewage sludge contains the second greatest amounts of this element. The only organic waste containing more phosphorus is bone meal, but on a global scale it is produced in much smaller quantities than sludge. Therefore, sludge is considered a very promising source of phosphorus (Cordell et al., 2011; Havukainen et al., 2016).

Phosphorus must be separated from the wastewater stream flowing out of the treatment plant, because too much phosphorus brought to the natural reservoirs could exacerbate eutrophication, which by stimulating the growth of algae and other photosynthetic organisms may ultimately lead to the formation of extinction zones in the sea, called the deserts of the sea. Such a phenomenon was first observed in a first half of 20th century (Ashley et al., 2011). Currently, the most widely used technology of biological treatment is conducting alternating aerobic and anaerobic processes, called enhanced biological phosphorus removal (EBPR). The first step is to carry out the anaerobic process, during which the organisms in the activated sludge hydrolyze polyphosphates and release phosphorus from cells in the form of orthophosphates, absorbing simple organic compounds from the environment and storing them in the form of poly-β-hydroxybutylic acid. Next, the activated sludge goes into an oxygenated chamber, where the bacteria which have the ability to store larger amounts of phosphorus that is necessary for their physiological needs begin to retain phosphorus, in the form of polyphosphate, in their cells (de-Bashan and Bashan, 2004; Xie et al., 2011; Zhou et al., 2016; Tarayre et al., 2016). This is a very wide range of organisms, including inter alia: *Aeromonas*, *Pseudomonas*, *Alcaligenes*, *Comamonas-Pseudomonas*, *Flavobacterium-Cytophaga*, *Moraxella*, *Xanthomonas*, *Paracoccus*, *Bacillus*, *Corynebacterium* and many other bacteria, mainly gram-positive (Bao et al., 2007). The process can be also enhanced by microalgae and other autotrophic organisms (Tarayre et al., 2016). With such a rich fauna and flora of the sludge, it can retain very large amounts of nutrients, including phosphorus. Scientists say that 90% of the phosphorus that goes to the treatment plant together with influent sewage is retained in the activated sludge (Schütte et al., 2015). Most of the phosphorus is stored in the flora cells of the activated sludge in the form of adenosine triphosphates and about 10% of the phosphorus is precipitated as iron and aluminum chlorides, with coagulants based on aluminum and iron (PIX and PAX) added to the process (Tan and Lagerkvist, 2011; Tarayre et al., 2016). The use of the above-described alternating anaerobic and aerobic process allows a high degree of concentration of phosphorus in the sludge (from 1% to 5%, and in extreme cases up to 15% of phosphorus in the dry matter of the residue) (Tao and Xia, 2007). Apart from the concentration of phosphorus in influent wastewater, which is mainly connected with the industrialization of the agglomerations from which sewage is collected, many other factors affect the ability of the sludge to retain phosphorus. These include parameters such as pH, temperature, COD, the degree of oxygenation, magnesium or organic fraction content, or even the age and condition of the sludge (de-Bashan and Bashan, 2004; De Lucas et al., 2007; Wachtmeister et al., 1997). Skillful control of those parameters makes it possible to increase the efficiency of phosphorus recovery by 50–143%. More efficient removal of phosphorus from treated sewage, aside from undeniable ecological advantages, is associated with improved efficiency of a phosphorus recovery process in the later stages of processing sludge, regardless of the technology used (de-Bashan and Bashan, 2004; Ehbrecht and Schuhmann, 2009; Kapagiannidis et al., 2009). The phosphorus removal technology improvement is worth considering since the European Water Framework Directive in 2000/60/EC states that the maximum allowable concentration of nitrogen and phosphorus in wastewater treatment plant effluents will change to respectively <2,2 mg N/L and <0,15 mg P/L in treated wastewater (Scherrenberg et al., 2009). Such an approach will be encouraged since some regions, like for example Baltic sea catchment area, are particularly vulnerable to eutrophication (Saktaywin et al., 2005). The newest trends in wastewater treatment assume separate collecting of urine and fecal waste with an implementation of separate treatment technologies. Nevertheless, such an approach requires implementation of such a concept on a city scale, not only the treatment facility scale. Still, an economical and ecological phosphorus recovery system will be a necessity in the nearest future (Ashley et al., 2011; Cordell et al., 2011; Cordell et al., 2009).

4. Methods of phosphorus recovery from sewage sludge

The simplest method of phosphorus recovery from sludge is the direct use of activated sludge as a fertilizer. However, just the transport and management of highly hydrated sludge (usually above 50% H_2O) can generate from 25 to 65% of the total operating cost of a treatment plant (Li et al., 2014; Xie et al., 2011). At the same time, sewage sludge can contain significant amounts of potentially hazardous organic contaminants, for example aromatic hydrocarbons and heavy metals. Legal regulations restricting the use of sewage sludge as fertilizers are increasingly strict, especially those defining the maximum allowable concentrations of heavy metals in the sludge introduced into the soil (Suciu et al., 2015; Zhou et al., 2016; Tarayre et al., 2016). For this reason, technologies for sludge treatment and indirect recovery of phosphorus are becoming increasingly popular.

Due to the introduction of new laws in European Union, landfilling of sludge which cannot be applied in agriculture has already been prohibited in most of the member countries. Therefore, the new technologies of processing sludge are developing faster and faster and are implemented in a growing number of sewage treatment plants, both small and large. It is important to realize that sewage sludge is often directly used as fertilizer, especially in third world countries, mainly because of a lack of access and funds to implement sewage sludge incineration and phosphorus recovery technologies (Biplob et al., 2009; Cordell et al., 2011). Sewage sludge is subjected to drying in order to increase the bioavailability of phosphorus since organic phosphorus fraction seems to be less bioavailable than the inorganic one (Li et al., 2014). Phosphorus recovery technologies which can be employed in treatment plants can be divided into two groups: recovery of phosphorus from sewage sludge and leachates, and phosphorus recovery from sewage sludge ashes. They will be described in detail in the following sections.

4.1. *Phosphorus recovery from sewage sludge and leachates*

At the moment, the most widely used technologies of phosphorus recovery are those based on precipitation of phosphoric minerals from sludge or leachates. Phosphoric minerals can be precipitated in the form of struvites, hydroxyapatites or calcium phosphates. It is a group of the best-known phosphorus recovery technologies, since they have already been implemented in many plants, mainly in Japan (Yuan et al., 2012). Their most important advantage is the ability to obtain high-quality phosphoric minerals, which can find a direct application in agriculture. Precipitated hydroxyapatite materials contain very low concentrations of heavy metals and are thus considered to be safe for the environment (Nakakubo et al., 2012). Furthermore, struvite is considered poorly soluble, which means that even in case of applying a vast amount of such fertilizer to the ground, the possibility of polluting the environment with a high load of phosphorus and enhancing the eutrophication process is low (Kataki et al., 2016; Shu et al., 2006). Another advantage of implementing such a technology is connected with the spontaneous struvite precipitation phenomenon. In some specific conditions, especially if a sewage sludge treatment plant runs the process of biogas recovery, struvite may cause clogging of the pipes. This problem occurs in most of such facilities when the Mg^{2+}, NH_4^+ and PO_4^{3-} concentrations are high and the pH is in the range $7.0 < pH < 10.7$. Precipitating compounds are only: struvite ($MgNH_4PO_4 \times 6H_2O$), amorphous calcium phosphate ($Ca_3(PO_4)_2 \times H_2O$), monetite ($CaHPO_4$), newberyite ($MgHPO_4$), calcite ($CaCO_{3(s)}$). Pipelines have to be maintained at least once every eight months, otherwise the facility can no longer function. An implementation of a struvite recovery system before a biogas recovery facility may solve this kind of a problem permanently (Jaffer et al., 2002; Lew et al., 2009; Le Corre et al., 2005). Moreover, the implementation of such a technology may enhance the N removal process, although only up to 12,5% (de-Bashan and Bashan, 2004).

However, taking into account the fact that these materials are obtained directly from sewage sludge or leachates, there is a possibility of introducing potentially hazardous organic pollutants or dangerous pathogens into the environment. The thus prepared materials must therefore be subjected to hygienization processes and possibly removal or neutralization of the organic fraction polluting the material. At the same time, a high concentration of organic matter could potentially inhibit the crystallization process by complexing calcium and occupation, by organic matter, of active sites on the surface of the crystals. A lot of parameters such as pH, COD (chemical oxygen demand), P, Mg and K content, temperature, time and intensity of oxygenation have a significant influence on the efficiency of the process (de-Bashan and Bashan, 2004; Havukainen et al., 2016). Moreover, conducting recovery from leachates does not guarantee a complete recovery of phosphorus (up to 40% recovery efficiency) or management of all process waste, including the remaining excess sludge (Biplob et al., 2009). What is more, the cost of producing 1 kg of phosphorus obtained that way is around 1 euro which is three times higher compared to regular fertilizer prices (Weigand et al., 2013). Other authors report that the operational cost of a struvite recovery installation is 50 000 £ per year, while the reimbursement could be at level of 16 000 to 20 000 £ per year (Jaffer et al., 2002). That is the reason why these technologies, despite the introduction of many improvements, are by some scientists considered incomplete, outdated and economically unjustifiable.

However, such a concept has an important advantage. Some authors claim that struvite may be recovered up to 97% if the Mg concentration in sewage sludge is sufficiently high. The best P:Mg ratio is 1:1.05, but on a technical scale it should generally be maintained at the level 1:1.3. The molar ratio of P:N has to be at least 1:1. Generally, sewage sludge does not contain sufficient amounts of magnesium, resulting in only 72% efficiency of the recovery process. In such cases $MgCl_2$, or another Mg source (wood ashes, magnesite, magnesia, bittern, seawater or by-products of MgO production) is often added, which obviously generates a cost increase (de-Bashan and Bashan, 2004; Jaffer et al., 2002; Pastor et al., 2008; Jordaan et al., 2009; Kataki et al., 2016). Fig. 1 presents a diagram of example concepts of phosphorus recovery from sludge or leachates, together with the concentrations of major pollutants occurring there. The process of recovery after fermentation may be performed before (31% of recovery efficiency) or after the thickening process (85% of recovery efficiency). Nevertheless, in such cases the pipe clogging problem would not be solved (Nakakubo et al., 2012; Pastor et al., 2008).

Some authors claim that struvite may be precipitated through a Biological Nutrient Removal Process before wastewater treatment, causing 83–85% of phosphorus recovery (Benisch et al., 2009). Others claim that struvite precipitation will never be satisfactorily efficient because of the high ammonia demand (Tao and Xia, 2007). However, there are concepts of phosphorus recovery from leachates and sludges based on nonapatite phosphorus fraction recovery. Drying of sewage sludge may lead to obtaining inorganic forms of phosphorus such as MgSiP2, NaAl(p2O7), SiP2O7 or AlSi2P3O12. Those can be subjected to hydrolysis in 3,5 bar pressure and 140 °C (KREPRO process) or sulphuric acid extraction (BioCon Process). Nevertheless, the obtained phosphorus material may still be contaminated with both heavy metals and organic pollutants, which will determine the need for implementing further processing of the obtained material (Li et al., 2014; Pettersson et al., 2008).

4.2. *Phosphorus recovery from the sewage sludge ashes from thermal disposal of sewage sludge*

According to the latest literature reports, phosphorus recovery in the near future should focus on the ashes from sewage sludge incineration. Currently, much more literature is published that addresses the issues of phosphorus recovery from thermal utilization of sludge than the recovery from sewage and leachates. Ashes from sewage sludge thermal treatment, mainly due to significant reduction (70–90%) in the volume of the incinerated materials, contain much higher amounts of phosphorus (Guedes et al., 2014; Biplob et al., 2009). The content of this element in sludge is estimated to be from 1 to 5% (up to 15% if an appropriate technology is used and with very high loads of phosphorus flow into the sewage treatment plant), while in ash the content is from 5 up to 11% (maximum concentrations of up to over 20%) (Ottosen et al., 2013; Weigand et al., 2013; Biplob et al., 2009). Some authors claim that the concentration of phosphorus in sewage sludge is around 0,12 g/L, while ashes after sewage sludge incineration may contain from 70 to 134 g/kg of the mentioned element (Couto et al., 2015). Even the ashes with an average amount of phosphorus contain about 8% of it, which corresponds to the content of medium phosphorus deposits. From 5 to 10 times more phosphorus can be recovered from the ashes than the sludge and leachates, but unfortunately such technologies are economically viable only in large sewage treatment plants, mainly because of the need to incur large capital expenditures associated with building a facility which meets all the ecological criteria for sludge incineration. These solutions are, however, already used, for example in the Netherlands (Cordell et al., 2011). It is important to mention that large-scale facilities process most of the sludge due to high processing capacity. For example in the Pomeranian voivodeship in Poland, two sewage sludge thermal treatment plants process over 60% of all sewage sludge produced in over 220 sewage sludge treatment plants.

Incineration of sewage sludge is most often carried out at a temperature of about 850° C. At this temperature, phosphorus takes a form of volatile oxides, which then condense upon cooling to a temperature of 400–600° C to form P_4O_{10}, becoming a component of the ash retained by the filters. When considering the co-combustion of sewage sludge (10% addition of sewage sludge to other fossil fuels) it is important to realize that such an approach may cause problems with SCR systems (Selective Catalytic Reduction). Moreover, conducting the phosphorus recovery is more efficient when ashes contain a higher concentration of phosphorus (Beck and Unterberger, 2006; Cieślik et al., 2014). Furthermore, the combustion of sewage sludge in 1250 °C, even without coal, results in a lower concentration of phosphorus in ash fraction and a higher concentration of phosphorus in dust fraction, which contains a variety of contaminants due to the mandatory exhaust gas treatment process (Zhang and Ninomiya, 2007). That is why the creation of separate facilities for sewage sludge incineration appears to be justified (Cieślik et al., 2014).

Typically, ashes contain more phosphorus than slag fractions or finer dust fractions, however if the incineration process is carried out in a fluidized bed, the slag fraction is never created, and there is a probability that the dusts can be used as a dopant for light building materials (Tan and Lagerkvist, 2011; Pettersson et al., 2008; Takahashi et al., 2001, Pavsic et al., 2014). Despite the often low pozzolanic activity of dust fraction, such waste can be stabilized together with ash fraction, especially if the ashes have been previously deprived of phosphorus. The main elements of ash fraction are: Ca, Si, Al, Fe, P and O. Most of them occur as oxides forming such compounds as CaO, SiO_2, Al_2O_3 or Fe_2O_3 (Takahashi et al., 2001). A high concentration of CaO and SiO_2, which are the main components in residues after phosphorus extraction, is the main reason why such waste could be used as a component of building materials (Biplob et al., 2009; Ottosen et al., 2013). Such an approach allows a complex management of all solid residues formed during sewage sludge incineration and phosphorus recovery processes, enabling the design of “no solid waste generation” technology.

In ash fraction, phosphorus often occurs as $Fe_4(P_4O_{12})_3$), $Al(PO_3)_3$, mainly because $AlCl_3$, $Al_2(SO_4)_3$, $FeCl_3$, $Fe_2(SO_4)_3$)

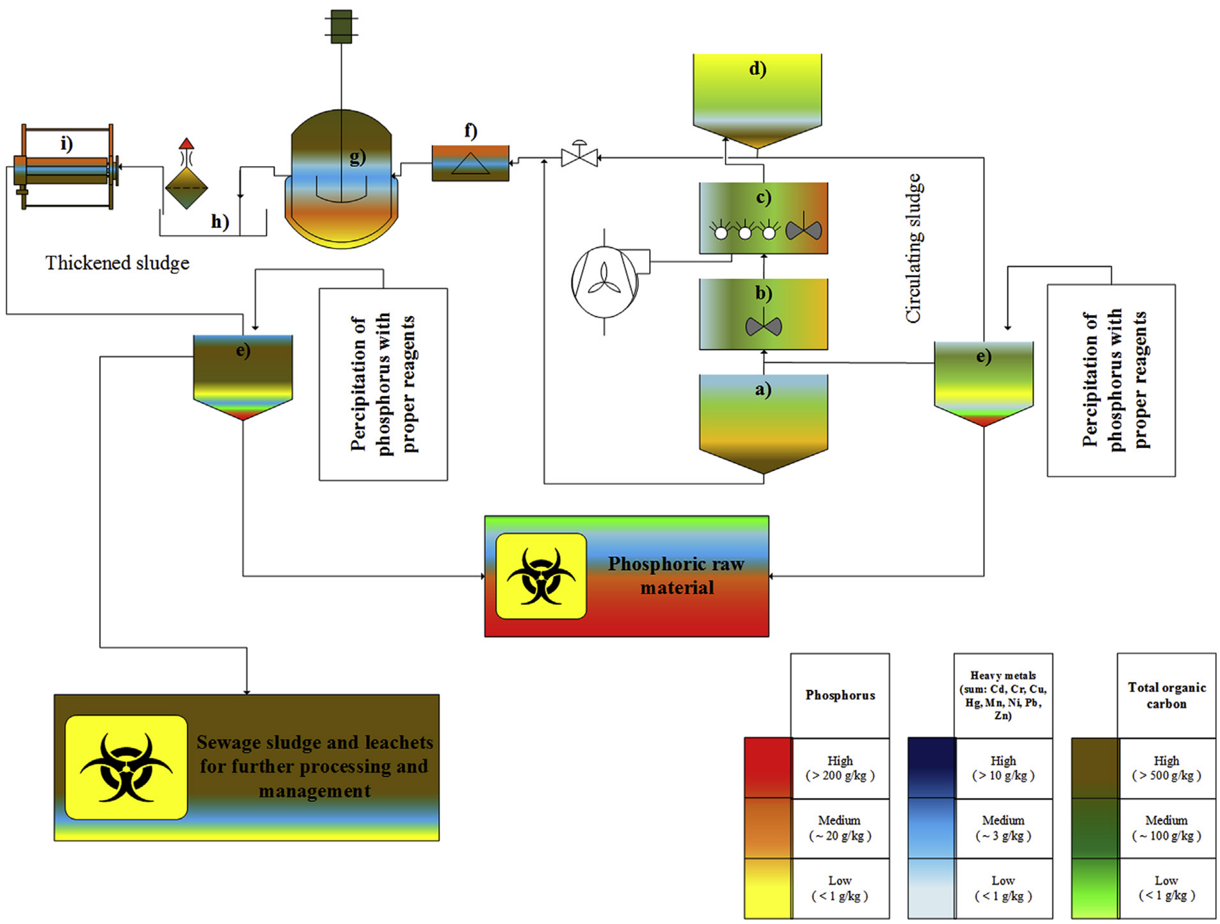

Fig. 1. Concept of phosphorus recovery from sewage sludge ashes with process residues management; a) sewage sludge silo, b) dryer, c) fluidized bed furnace, d) heat exchangers, e) filters, f) exhaust gases treatment reactor, g) chimney, h) ashes silo, i) dust silo, j) phosphorus recovery reactor, k) facility for processing of solid residues (Donatello and Cheeseman, 2013; Li et al., 2015; Lapa et al., 2007; Nakakubo et al., 2012; Garrido-Baserba et al., 2014; Cordell et al., 2011; Rodríguez et al., 2013).

compounds are widely used as phosphorus precipitating agents (Biplob et al., 2009; Pettersson et al., 2008; Ottosen et al., 2013). It seems that if Al based compounds are used for phosphors precipitation, the extraction is 10–95% more efficient. Nevertheless, a high concentration of an Al compound may cause a greater threat for the environment than high concentrations of Fe based compounds. (Pettersson et al., 2008). However, acidic extraction processes are not the only ones available on the market.

Some approaches assume direct use of ashes as a phosphorus source or fertilizers. During sewage sludge combustion the vast majority of potentially hazardous organic compounds, as well as all parasites and pathogenic microorganisms are completely neutralized (Pettersson et al., 2008; Guedes et al., 2014). In such a case it is easy to implement a variety of thermochemical treatment methods with creation of volatile chlorides to remove heavy metals fractions. The process is carried out in 900–1050 °C with 46 kg/t_{ash} NaCl, 39 kg/t_{ash} MgO and 49 kg/t_{ash} NaHCO3 as additives and leads to obtaining 13–18% of P_2O_5 in final product. There is a possibility to remove up to 90% of Cu and Zn, along with 95% of Cd and Pb, from the ashes fraction (Nowak and Winter 2013; Havukainen et al., 2016). However, the process is not capable of removing Cr or Ni. A situation in which concentrations of even a few heavy metals exceeds the legal limits may disqualify the application of the obtained material in agriculture (Vogel et al., 2014; Havukainen et al., 2016). Nonetheless, some authors claim that the ashes obtained during sewage sludge incineration above 700 °C are unsuitable for use as fertilizers, mainly due to the biounavailable form of phosphorus compounds occurring in the mentioned materials (Ottosen et al., 2013; Guedes et al., 2014; Nakakubo et al., 2012; Pettersson et al., 2008; Yuan et al., 2012). Moreover, all N compounds are volatilized as NO_x, which also results in a decrease of fertilizing qualities of the obtained material. Therefore, the implementation of expensive phosphorus extraction or other processing methods is often desired (Yuan et al., 2012).

Neverheless, phosphorus is most often obtained from the ashes through extraction using mineral or organic acids such as H_2SO_4, HNO_3, HCl, H_3PO_4 citric or oxalic acids. Sulphuric acid extraction is considered the least expensive process, while H_3PO_4 extraction is considered the most expensive. Moreover, an extraction using sulphuric acid results in leaching of minor amounts of heavy metals, due to the fact that fewer complexation reactions occurs. Even in the case of the high Pb concentration, this element is extracted in amounts three times smaller than when using HCl as the extraction medium. Moreover, HCl can decrease the quality of the construction material if the process residues are assumed to be used as a dopants (Takahashi et al., 2001; Pettersson et al., 2008; Ottosen et al., 2013). In the case of SEPHOS process, the most suitable pH for the extraction is 1.5. Recovery of almost 100% of phosphorus is achievable in 2–4 h time, together with 90% of Ca and 65% of Al. Aluminum, cadmium and other heavy metal fractions, extracted in high amounts during such a long process, may inhibit plant growth, cause necrosis and long term environmental pollution in the case of using the obtained material as fertilizer, although pH decrease may result in P recovery increase and Cd extraction decrease (Nakakubo et al., 2012). Usually the plato of extraction process is obtained in less than 100 min. The process efficiency does not depend on the temperature, but a growth of temperature may increase the process speed. The main ingredients of the obtained liquid extract are P, K, Ca, Mg and S. The thus obtained liquid phosphorus material can be used not only as a fertilizer, but also in industry, for example pharmaceutical (Tan and Lagerkvist, 2011; Biplob et al., 2009). If the concentrations of heavy metals in the extracted media are high, it is also possible to precipitate this impurity fraction using caustic soda. Direct extraction using NaOH is also possible, but the efficiency of such a recovery process reaches only 40% (Biplob et al., 2009; Nakakubo et al., 2012).

More and more phosphorus recovery technologies are being created. Among them it is worth mentioning electro dialysis with 32–84% recovery, which is described by (Couto et al., 2015; Guedes et al., 2014). During the mentioned process gypsum fraction is also created, so further studies should be made to assess the possibility of using process residues as dopants for construction materials. Other methods of phosphorus recovery from ashes, which are becoming more popular due to increasingly broad dissemination of the technology for thermal disposal of sewage sludge. Emerging regulations prohibit landfilling of sewage sludge, furthermore solid residues after sludge incineration should also be managed in an ecologically and economically viable manner. As mentioned before, according to some reports in the literature, it is possible to use residues from phosphorus extraction as an additive to building materials (Ottosen et al., 2013). As a result, it is very likely that a technology can be designed that will allow not only the recovery of valuable phosphoric material, but also comprehensive management of all solid waste generated during sewage sludge thermal treatment. In such a case ash fraction has to be processed to recover liquid phosphorus fraction. After the phosphorus extraction process the acidic solid residues fraction have to be separated. A the same time solid residues after exhaust gas treatment that arise during sewage sludge incineration are separated at the second set of filters. Both fractions can be mixed with a stabilizing agent and cemented, which would result in a production of construction materials. In such a case all waste produced in both the sewage sludge thermal treatment and the phosphorus recovery process would be managed in an economically and ecologically justifiable manner. Fig. 2 shows a concept of phosphorus recovery from ashes, together with the concentrations of major pollutants at different stages of the process of sewage sludge thermal treatment and acidic extraction of phosphoric material.

5. Analytical side of phosphorus recovery

When designing an ecological and economical method of phosphorus recovery in a wastewater treatment plant, the information about the concentration of phosphorus in the resulting material is not sufficient to determine the usability of the material produced. Phosphorus, both in sewage sludge and in the ash after its incineration, can occur in various forms. During the precipitation of struvite or hydroxyapatites, various other mineral fractions are obtained.

Different types of calcium compounds are often a part of the precipitated phosphoric raw materials and process residues after extraction. They may be, inter alia, CaO, $Ca(OH)_2$, $CaSO_4$, $CaCO_3$, ($Ca_3(PO_4)_2 \times H_2O$), or $CaHPO_4$ (de-Bashan and Bashan, 2004; Lew et al., 2009). The ideal technique which can be used to determine the content of the individual mineral fractions is spectroscopy with X-ray diffraction (XRD). This method also makes it possible to carry out speciation analyses.

An example of this may be the determination of the content of chromium in its +3 and + 4 state of oxidation. It is then possible to estimate the actual toxicity of the sample, since, as is known, chromium compounds in +4 and + 6 oxidation state are characterized by much higher toxicity than compounds of chromium in +3 state (Vogel et al., 2014). The XRD technique is also used in controlling of the reaction of phosphorus recovery to determine the desired minerals: struvite ($MgNH_4PO_4 \times 6H_2O$) or hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$).

Very useful in observing the structure and thereby estimating the quality of the produced crystals are the techniques of scanning electron microscopy (SEM) (Tan and Lagerkvist, 2011; Schütte et al., 2015) and transmission electron microscopy (TEM). TEM makes it possible to observe the formation of gypsum during the extraction process. This fraction is a part of the solid post-extraction residues. This mineral is well suited as an additive to construction materials (Zhang and Ninomiya, 2007; Ottosen et al., 2013). Because of that, solid post-extraction residues can be made into light construction material, e.g. when mixed with dust fraction. As mentioned earlier, such an approach enables the management of all solid waste generated during processing and management of sewage sludge.

Determining all the major components of the tested materials is necessary both in the case of the manufactured building materials and the phosphoric raw materials in order to assess their usability. For this purpose, the best technique is spectroscopy with X-ray fluorescence (XRF). This technique allows conducting analyses without the need to destroy the test sample, making it possible to perform routine inspections of the manufactured materials. With the use of XRF one can determine many mineral molecular entities, including heavy metals. It is their concentration that is often a limiting factor in the use of the material, for environmental reasons (De Lucas et al., 2007; Pettersson et al., 2008; Takahashi et al., 2001; Weigand et al., 2013; Ottosen et al., 2013; Zhang and Ninomiya, 2007; Vogel et al., 2014).

Many heavy metals are poorly soluble in water, but even small amounts of pollutants leaching from the tested materials could pose a threat to the environment. If their concentration in the aqueous extracts is high, it is usually sufficient to use atomic absorption spectrometry (AAS) for determination (Takahashi et al., 2001). More often, however, it is used for determining the given elements in mineralized materials, which obviously manifest higher concentrations of analytes.

For this purpose X-ray photoelectron spectroscopy (XPS) is also effective, though less frequently used. It applies especially in the determination of entities of less than 1 μm in grain size (Zhang and Ninomiya, 2007; Takahashi et al., 2001). Among more modern techniques for the determination of metals in samples and extracts of the tested materials is the technique of atomic emission spectrometry with inductively coupled plasma (ICP-AES) (Vogel et al., 2014; Guedes et al., 2014; Schütte et al., 2015; Zhang and Ninomiya, 2007; Biplob et al., 2009; Couto et al., 2015). The ICP-AES technique may also be applicable in the determination of phosphorus, but for unknown reasons literature reports rarely mention such usage.

UV–visible spectrophotometry with the use of molybdenum standard is the most frequently used method of phosphorus determination. The determination is carried out on the basis of European standards like EN ISO 6878. Typically, a large part of phosphorus from acidic extraction is in the form of phosphates. Unfortunately, the range of phosphate content in the recycled material is very wide (between 5 and 95%), but still the technique of ion chromatography (IC) may prove useful in the determination of those entities in order to assess the quality of the produced material. Thanks to the use of IC it is possible to simultaneously determine many anions (SO_4^{2-}, NO_3^-, Cl^-, F^-) and cations (NH_4^+, Na^+, Mg^{2+}, K^+) in one analysis and thus the usability and the ecological

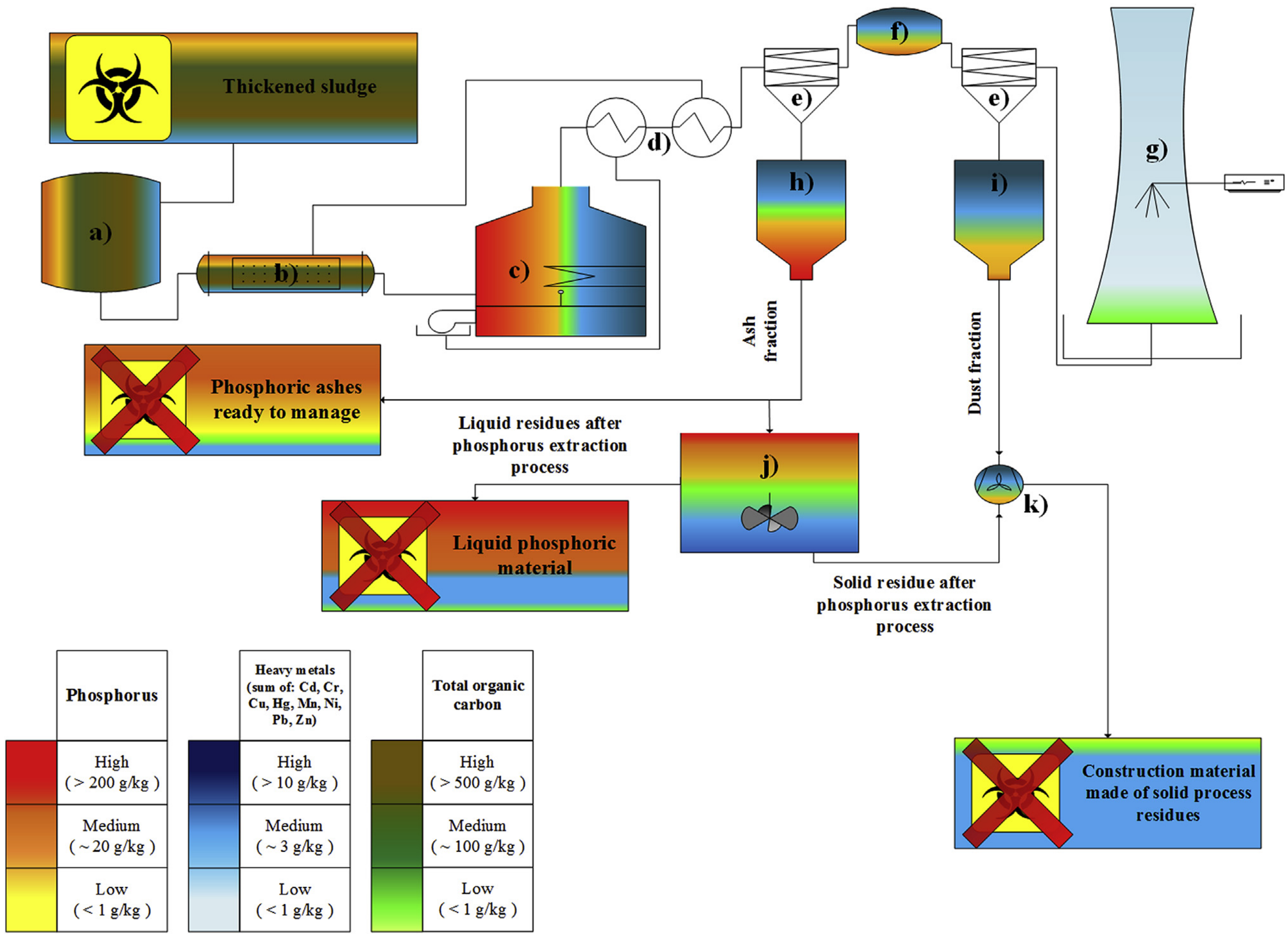

Fig. 2. Concept of phosphorus recovery from sewage sludge ashes with process residues management; a) sewage sludge silo, b) dryer, c) fluidized bed furnace, d) heat exchangers, e) filters, f) exhaust gases treatment reactor, g) chimney, h) ash silo, i) dust silo, j) phosphorus recovery reactor, k) facility for processing solid residues (Donatello and Cheeseman, 2013; Li et al., 2015; Lapa et al., 2007; Nakakubo et al., 2012; Garrido-Baserba et al., 2014; Cordell et al., 2011; Rodríguez et al., 2013).

safety of the tested materials can be decided (Pastor et al., 2008; Pettersson et al., 2008). These analyses are performed in accordance with EN ISO 11885 and EN ISO 10304. However, a single analysis is very time, money and labor consuming. Due to the aforementioned reasons it is not possible to conduct phosphorus determination at every single step of phosphorus recovery. Phosphorus nuclear magnetic resonance spectroscopy (P-NMR) with capability to perform an on-line analysis seems to be the best available technique for such a purpose (Ehbrecht and Schuhmann, 2009; Li et al., 2014). Fig. 3 presents a summary of analytical techniques which can be used during inspections of the phosphorus recovery processes and possible further management of sewage sludge.

6. Discussion on benefits of implementing phosphorus recovery technology in sewage treatment plants

Implementation of a phosphorus recovery technology in the majority of sewage treatment plants in the world would allow a normalization of prices of chemical fertilizers and facilitate the rational management of phosphorus. Also, many countries that do not have large natural deposits of phosphorus could become, in this matter, independent from the previously mentioned leaders in that field, such as the US, China and Morocco. It is likely that a broad implementation of such a technology could prevent some economic conflicts in the future (Couto et al., 2015; Guedes et al., 2014).

One must not forget about the widespread problem of world hunger. Paradoxically, Africa, struggling with the biggest problem of hunger, exports the largest quantity of phosphoric raw material. Worldwide, approximately 30 million tons of these materials are transported annually, which is inseparably associated with the huge consumption of power media and pollution emission. The implementation of phosphorus recovery technology in sewage treatment plants worldwide would have an indirect influence on reducing the amount of pollutants emitted. Even an increase in demand for biofuels may indirectly affect the global demand for phosphorus, because it is necessary for the efficient production of large quantities of biomass (Cordell et al., 2009).

The high demand for fertilizers generates the need for exploiting low grade deposits containing the aforementioned impurities such as heavy metals and radioactive elements. Ashes obtained after sewage sludge incineration generally contain less heavy metals like Cd, Cr, Hg, V and U than Morocco phosphorus ores. Sewage sludge ashes, however, contain more Cu, Ni, Pb and Zn than mentioned ores, but in every region those ratios can differ (Weigand et al., 2013). Concentrations of heavy metals, phosphorus and other elements in sewage sludge and sewage sludge ashes are presented in Figs. 4–9. The data were collected from 30 European and Asian wastewater treatment plants (Kataki et al., 2016; Zhou et al., 2016; Havukainen et al., 2016; Tan and Lagerkvist, 2011; Nowak and Winter 2013; Vogel et al., 2014; Takahashi et al., 2001; Weigand et al., 2013; Guedes et al., 2014; Ottosen et al.,

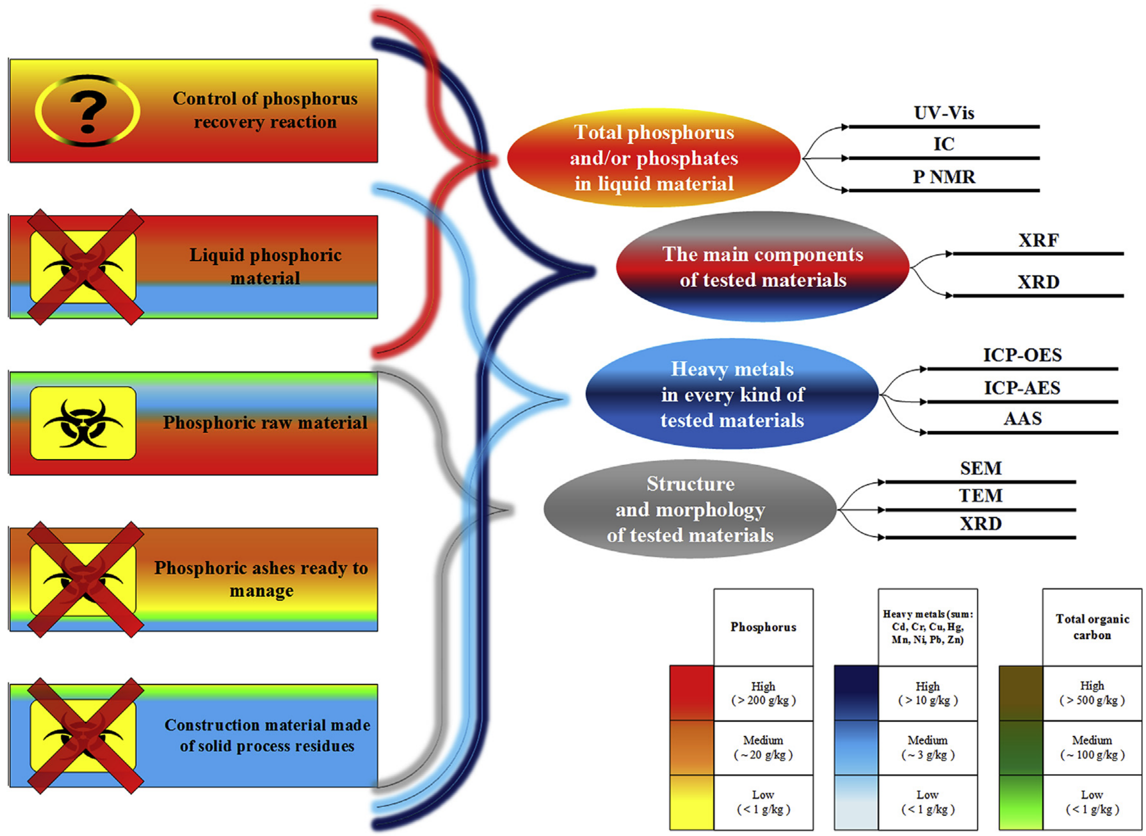

Fig. 3. Main analytical techniques and their usage during the processes of phosphorus recovery and process residues management (Ehbrecht and Schuhmann, 2009; Vogel et al., 2014; \Nowak and Winter, 2013; Biplob et al., 2009; Pettersson et al., 2008; De Lucas et al., 2007; Ottosen et al., 2013).

2013) (Pettersson et al., 2008; Liu et al., 2011; Lag-Brotons et al., 2014; Houillon and Jolliet, 2005; Rodríguez et al., 2013; Nakakubo et al., 2012).

Production of good quality fertilizers from low grade, contaminated ores inevitably involves generating potentially hazardous waste fractions. During the processing of minerals into phosphate fertilizers, a major waste occurring in large quantities is phosphogypsum, which contains significant quantities of heavy metals and potentially dangerous amounts of radioactive elements, and thus cannot be re-used (Pettersson et al., 2008; Weigand et al., 2013).

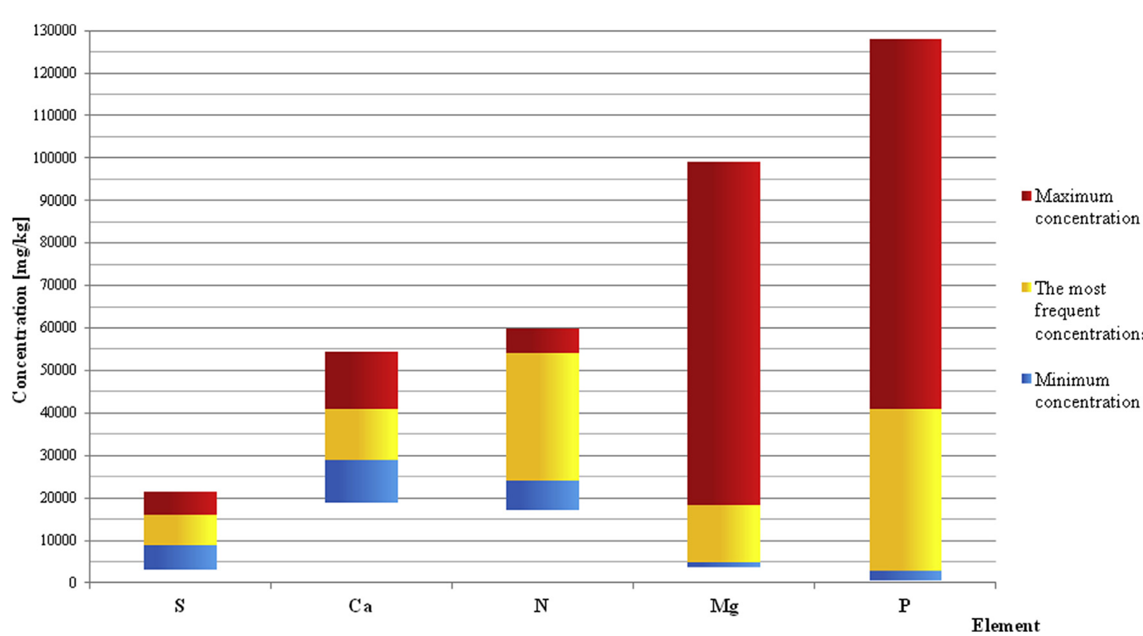

Fig. 4. Concentration ranges of major elements in sewage sludge from European and Asian wastewater treatment plants.

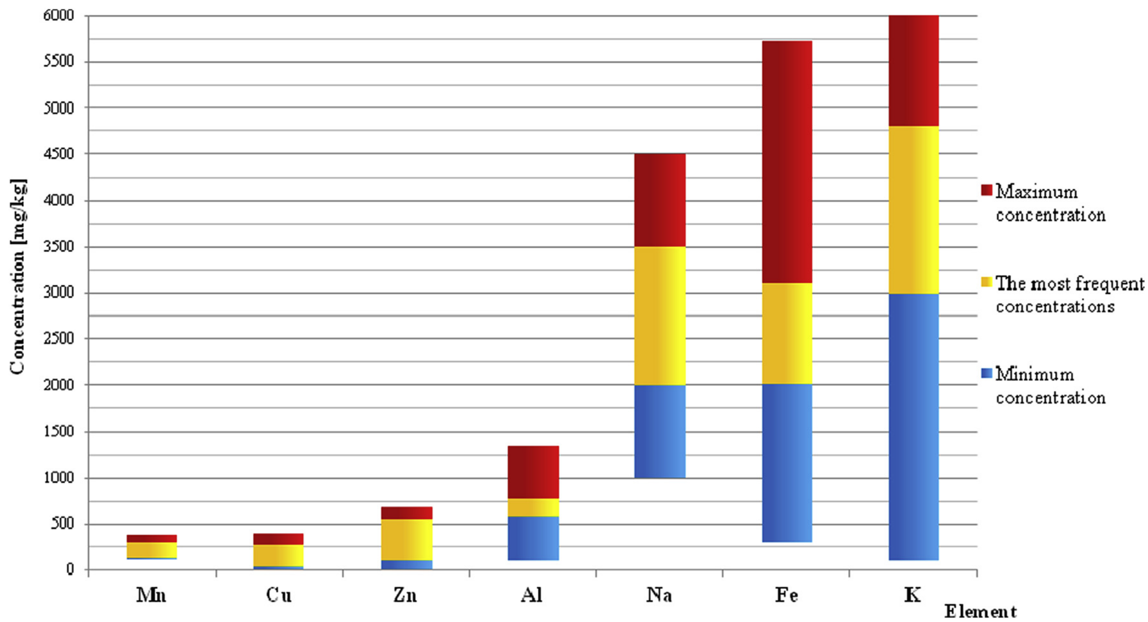

Fig. 5. Concentration ranges of elements in medium amounts in sewage sludge from European and Asian wastewater treatment plants.

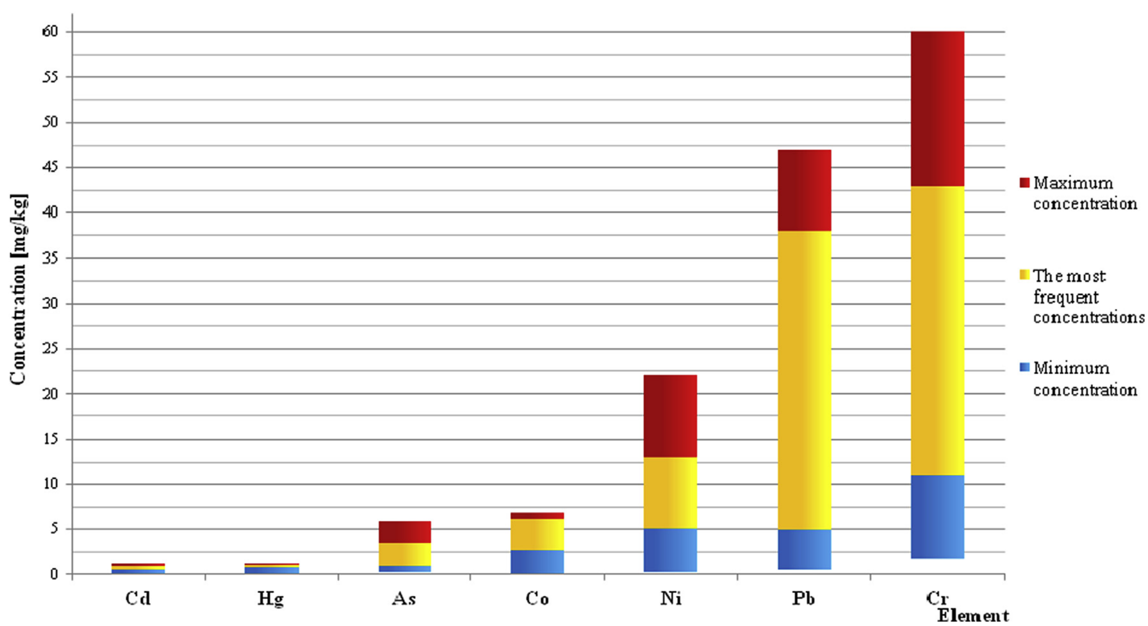

Fig. 6. Concentration ranges of minor elements in sewage sludge from European and Asian wastewater treatment plants.

Producing fertilizers and other phosphoric materials from processed sewage sludge which does not contain such high concentrations of these impurity fractions, results in materials of required quality without having to implement expensive technologies of purification of the end products. At the same time, the process waste does not include significant amounts of impurities, making it possible to search for alternative solutions for comprehensive management of all the waste generated during the process. Moreover, as shown in Figs. 7 and 8, ashes contain relatively high concentration of elements like K, Mg, Na and P. This also proves the high quality of ashes as a raw material for fertilizers production. Furthermore, concentrations of highly harmful elements such as: Hg, As, Co, Cd, Ni, Cr are at trace level in most of the examined sewage sludge treatment plants. Such a situation is promising if phosphorus recovery methods from ashes are going to be evaluated or even implemented.

According to some scientists, 15–20% of the global demand for phosphorus can be satisfied by recovering this element from sewage sludge (Yuan et al., 2012). It is estimated that widespread use of the mentioned technologies in Germany alone could satisfy 40% of the agricultural and industrial demand for phosphorus in that country (Weigand et al., 2013). In highly developed countries, such as Japan, recovery of phosphorus in wastewater treatment plants is standard procedure (Nakakubo et al., 2012; Takahashi

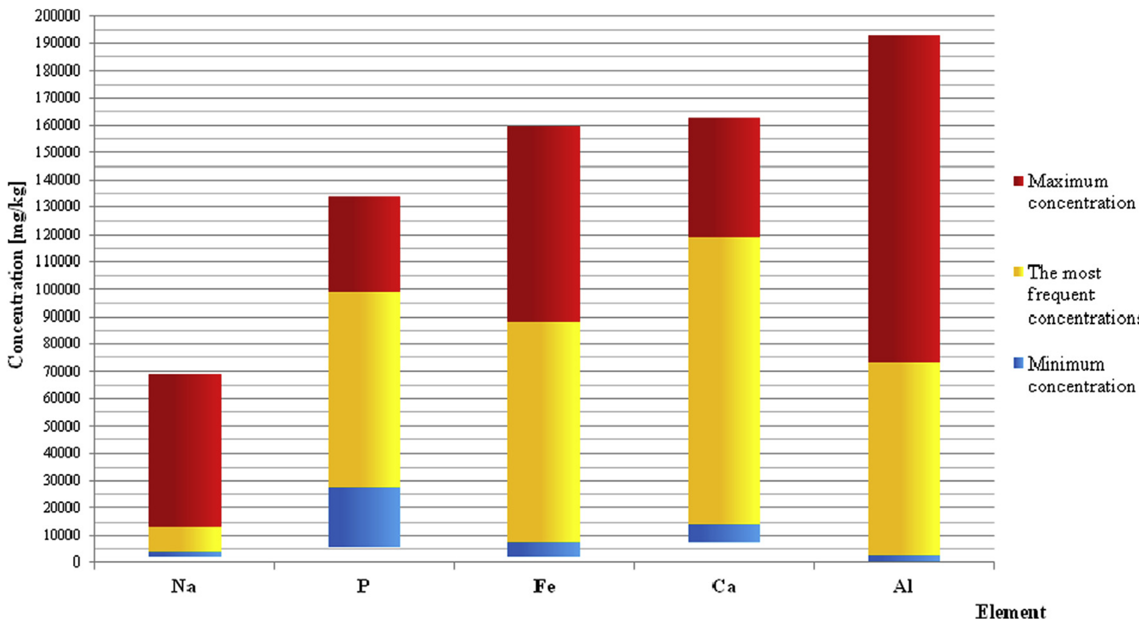

Fig. 7. Concentration ranges of major elements in ash fraction obtained after sewage sludge incineration from European and Asian wastewater treatment plants.

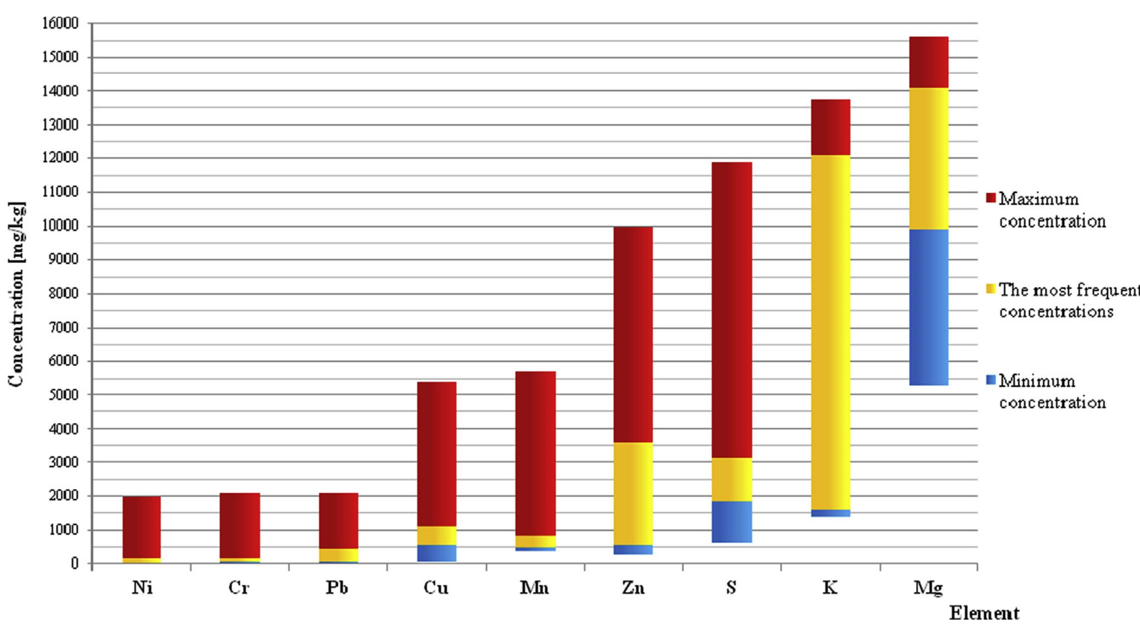

Fig. 8. Concentration ranges of elements in medium amounts in ash fraction obtained after sewage sludge incineration from European and Asian wastewater treatment plants.

et al., 2001; Yuan et al., 2012). As mentioned before, there are more than 30 ways of recovering phosphorus at various stages of wastewater treatment and sewage sludge management. These technologies, both for environmental and economical reasons, will be gradually implemented in treatment plants around the world. Industry and waste management will develop in the direction of low emission and recyclability. For this reason, establishments such as sewage treatment plants will be gradually changed into recovery facilities and the approaches concerning phosphorus recovery allowing an implementation of "no solid waste generation" technologies will be desired (Nakakubo et al., 2012). Advantages and disadvantages of the described concepts of phosphorus recovery are described in Table 1.

7. Summary

As early as since 2000 the EU Member States have been debating the implementation of more stringent standards for phosphorus removal from the treated effluent and the imposition of an obligation to recover phosphorus at one of the stages of either wastewater purification or treatment of excess sewage sludge. However, specific provisions have to be made in the field of phosphorus recovery from waste. Especially restrictions on heavy metals concentrations in waste materials which are going to be subjected to phosphorus recovery have to be evaluated and established. According to Life Cycle Analyses (LCA) conducted by researchers, the recovery of phosphorus from sewage sludge seems to be much more environmentally friendly than the direct use of sludge as a

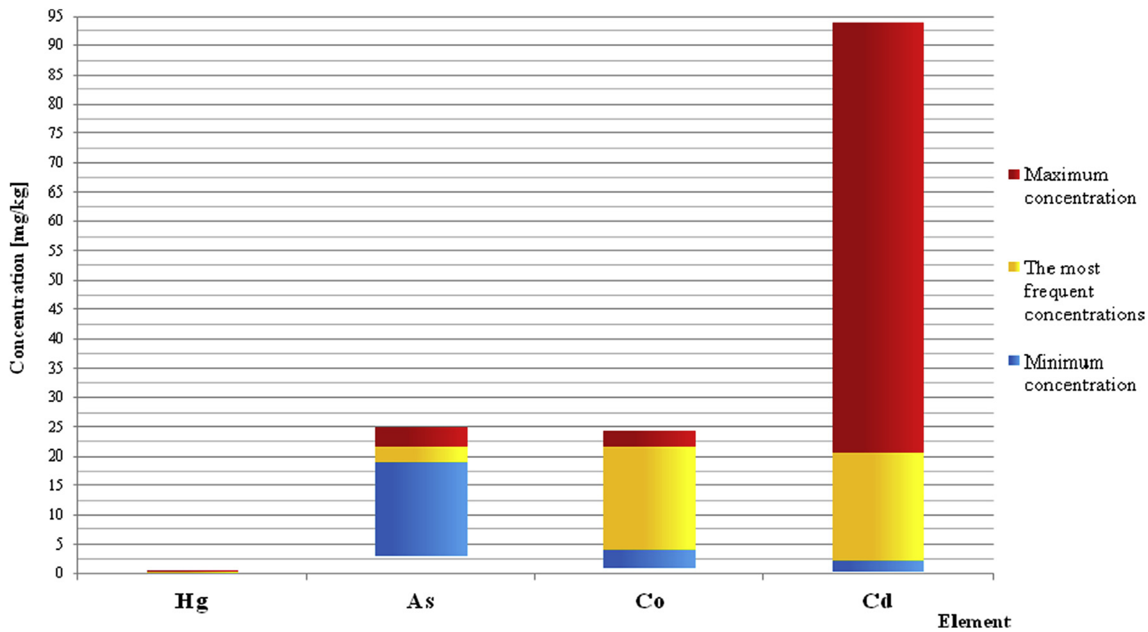

Fig. 9. Concentration ranges of minor elements in ash fraction obtained after sewage sludge incineration from European and Asian wastewater treatment plants (Kataki et al., 2016; Zhou et al., 2016; Havukainen et al., 2016; Tan and Lagerkvist, 2011; Nowak and Winter 2013; Vogel et al., 2014; Takahashi et al., 2001; Weigand et al., 2013; Guedes et al., 2014; Ottosen et al., 2013) (Pettersson et al., 2008; Liu et al., 2011; Lag-Brotons et al., 2014; Houillon and Jolliet, 2005; Rodríguez et al., 2013; Nakakubo et al., 2012).

Table 1
Advantages and disadvantages of implementing the selected phosphorus recovery methods in a sewage sludge treatment plant (Cieślik et al., 2014; Benisch et al., 2009; De Lucas et al., 2007; Havukainen et al., 2016; Jaffer et al., 2002; Kataki et al., 2016; Zhou et al., 2016; Tan and Lagerkvist, 2011; Shu et al., 2006; Nowak and Winter 2013).

Methods of phosphorus recovery	Possible unit processes applied to perform phosphorus recovery	Disadvantages of the method	Advantages of the method	Main groups of potentially hazardous pollutants
Direct use of sewage sludge in agriculture	Composting and stabilization in ponds Stabilization using earthworms Drying and pellet production	A relatively long stabilization time if low -temperature processes are used Possibility of contamination of the environment with a variety of organic pollutants, parasites and pathogens Applications limited to fertilizers and soil remediation Methods based on soil remediation not recommended by the European Union	Low investment costs Possibility of managing all sludge in case of small amounts of excess sludge If earthworm stabilization is used, there is low energy expenditure and a reduction in concentrations of heavy metals is possible Processes are cost-efficient even with small amounts of excess sludge	High organic carbon load Aromatic hydrocarbons Halogenated organic compounds Heavy metals Pathogens and parasites
Recovery from sewage sludge and leachates	Precipitation of phosphorus in form of struvite, hydroxyapatite	High investment costs Possibility of contamination of the environment with a variety of organic pollutants, parasites and pathogens Applications limited to fertilizers Incomplete phosphorus recovery (high ammonia and magnesium demand) Incomplete management of sewage sludge Incurred sewage sludge management costs are not fully recovered	Partial refund of costs Low probability of releasing heavy metals Slow phosphorus release Possibility of solving the problem of clogging pipes Increased nitrogen removal efficiency	Pathogens and parasites Organic pollutants
Recovery form ashes after sewage sludge incineration	Incineration Acidic extraction Thermochemical treatment Cementing	The highest investment costs Possibility of contamination of the environment with a variety of heavy metals and some organic pollutants Complicated processes Problems with obtaining high strength of the produced building materials Processes are cost-efficient only with large amounts of excess sludge	Partial refund of costs Considerable savings associated with waste disposal Complete management of sewage sludge High phosphorus recovery efficiency Possibility of simultaneous treatment of some heavy metals Wide range of applications Possibility of energy recovery during incineration process Less odours	Heavy metals Chlorinated species Halogenated organic compounds
No phosphorus recovery	Disinfection and chemical stabilization Incineration Vitrification Solidification of materials	Incomplete management of sewage sludge Generally connected with landfilling which is not recommended by the European Union Incurred management costs are not recovered High threat for the environment	Simple methods Less restrictive standards as compared to other methods	High organic carbon load Aromatic hydrocarbons Heavy metals Phosphorus Halogenated organic compounds Chlorinated species

fertilizer. Technologies based on phosphorus recovery from sewage sludge and leachates, such as struvite and hydroxyapatite recovery, gained popularity more than 10 years ago. However, such an approach allows to use the recovered material only as fertilizer and now it is giving way for more modern, comprehensive and ecologically friendly concepts. Technologies based on phosphorus recovery from ashes are gaining popularity faster and faster, mainly for socioeconomic reasons. Installations of sewage sludge thermal treatment are, for environmental reasons, becoming standard in the emerging plants that process large amounts of sewage sludge. Incineration in fluidized bed furnaces and separate collecting of ashes and dust fraction seems to be the most environmentally friendly and economically reasonable method. It is, however, important to select the most suitable method of phosphorus recovery for every single facility. More and more pressure is being put on the implementation of non-waste technology and materials recovery. This will result in a growing interest of "no solid waste generation" management concepts. Moreover, legislation pushing industry to implement "no waste" technologies have to be created. Technologies based on the recovery of phosphorus from sewage and leachates, despite the relatively low investment and maintenance costs, are not capable of complete management of all the waste generated during the operation of the plant. Thanks to the combined processes of sludge incineration, phosphorus recovery, and solidification of process residues and using them as a building material, it is possible to manage all streams of solid waste from wastewater treatment plants. Nonetheless, it is important to realize that, every sewage sludge treatment plant collect wastewater from different agglomerations. Wastewater reaching the plants may vary from each other significantly. In some cases, when for example sludge contains low phosphorus concentrations, implementing phosphorus recovery methods seems to be pointless. Dust and ash fraction can also differ significantly between sewage sludge treatment facilities. Even exhaust gases treatment technology can affect the quality of obtained dust fraction. In some cases implementing comprehensive management method, which is presented in the manuscript, would be impossible due to the characteristics of solid wastes produced during thermal utilization of sewage sludge. That is why every implementation of novel technologies should be considered separately for every sewage sludge treatment plant. Moreover, technologies of Cu, Ni, Pb and Zn separation from ashes or liquid phosphoric extract have to be investigated in the nearest future to allow a wide implementation of a phosphorus recovery process. The recovery of phosphorus in sewage treatment plants will be gaining in popularity with the increase in legislative pressure on the recovery of raw materials, reduction of phosphorus use and an increase in its prices. Even an increase in the demand for biofuels may indirectly affect the global demand for phosphorus because it is necessary for the efficient production of large quantities of biomass. Unfortunately, no organization has yet been established which would deal with the world phosphorus crisis. However, one can see a clear need for the implementation of meaningful resolutions concerning the management of phosphorus, otherwise a number of political conflicts, wars and the intensification of the problem of world hunger may happen in the coming decades.

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