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Coagulation of greywater from a small household

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ABSTRACT

The authors looked into developing a low cost technology for a treatment of greywater from a small household. The process of coagulation and oxidation with hydrogen peroxide, combined with ultraviolet radiation (H_2O_2 / UV), was investigated however, the article focuses only on coagulation and its effects. Since a highly concentrated greywater was used, doses of coagulant (poly-aluminium chloride) ranged from 12.5 to 200 g Al^{3+}/m^3. Coagulation with coagulant doses within the range of 25–100 g Al^{3+}/m^3 showed the best removal of turbidity, permanganate value (PV), chemical oxygen demand (COD) and total organic carbon (TOC). The lowest concentrations of residual aluminium (about 0.1 g Al^{3+}/m^3) were found during coagulation with the doses of 25–50 g Al^{3+}/m^3, while at the coagulant dose of 200 g Al^{3+}/m^3 the residual aluminium concentration was 1.4 g Al^{3+}/m^3. The lowest turbidity (1 NTU) was observed at the doses of 50–100 g Al^{3+}/m^3 and the Zeta potential from –10 to +14 mV. At the doses below 50 g Al^{3+}/m^3 or above 100 g Al^{3+}/m^3 the turbidity values were higher. The lowest concentration of residual aluminium corresponded to the Zeta potential from –10 to +14 mV. At the coagulant doses below 50 g Al^{3+}/m^3 or above 100 g Al^{3+}/m^3 the turbidity values were higher. The lowest residual aluminium concentration corresponded to the Zeta potential from –14 to –10 mV. The study helped to develop a mathematical model for coagulation. The model showed that coagulation of greywater (COD = 1,200 g O_2/m^3) at the coagulant dose of 100 g Al^{3+}/m^3 ensured about 63% process efficiency.

Keywords: Greywater; Coagulation; Zeta potential; Model of coagulation; Aluminium concentration; PV; COD; TOC

1. Introduction

Production of water for communal purposes becomes more and more costly so some decisions about its economic management have to be made. As it has been shown, reuse of greywater may be profitable, especially in areas where a water deficit is observed [1–5]. Water reclaimed from greywater can be used in agriculture (irrigation), for flushing toilets, car washing, in construction works or in some industrial installations for e.g. wet dedusting, cooling (heat exchangers, cooling towers), etc.

Treatment of greywater can involve various unit processes: physical, chemical and biological ones [1,5–14].

The main physical methods include: filtration on sand, gravel, charcoal, lime pebbles and natural zeolite, filtration through cartridge filters in a pipe installation, microfiltration, ultrafiltration, nanofiltration, reverse osmosis and adsorption.

The main chemical methods include: coagulation, electrocoagulation, chlorination, oxidation with hydrogen peroxide combined with UV radiation (H_2O_2/UV), photocatalytic oxidation (TiO_2/UV), (O_3/UV) and oxidation with the Fenton process with modifications.

The main biological methods include: membrane bioreactors, biological aerated filters, sequencing batch bioreactors, rotating biological contactors and anaerobic filters.

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The above unit processes show a different, usually rather low, efficiency in removing predominant organic pollutants from wastewater. In order to improve the overall process performance a sequence of several unit processes has to be employed [5,8,10].

The composition of greywater varies greatly with regard to both type and concentration of pollutants [3–5,7–9,11,15], for e.g. chemical oxygen demand (COD) can range from 100 to 2,000 g O_2/m^3, turbidity from 20 to 2,000 NTU, total organic carbon (TOC) from 30 to 600 g C/m^3 and pH from 6.3 to 8.5. Such high variability of the greywater characteristic makes it difficult to develop one universal treatment technology.

Currently, many different types of coagulants are available on the market and used in water and wastewater treatment. These are substances based on aluminium and iron compounds. Aluminium sulphate ($Al_2(SO_4)_3$ $18H_2O$) is still used as well as pre-hydrolysed poly aluminium chlorides such as PAX, Flokor and others. Pre-hydrolysed poly aluminium sulphates VI and poly ferric sulphates III (e.g. PIX) as well as poly aluminium and poly ferric chloro-sulphates are also produced. Poly aluminium chlorides may be used in water and wastewater treatment since they are very successful in the removal of pollutants from water and wastewater compared with coagulants that were not subjected to an initial hydrolysis [16]. They also offer better results than poly ferric coagulants [17] and are less sensitive to changes of water and wasterwater temperature and pH [16].

There are many monomeric and polymeric forms with positively charged hydroxy-aluminium groups in poly aluminium chloride solutions [16,18]. The popular belief is that polymers with 13 aluminium atoms (Al_{13}) remain stable in solutions of poly aluminium chlorides for several months at 298 K and at an alkali value $r = 2.4$ ($r = [OH^-]/[Al^{3+}]$) [19]. Poly aluminium chloride stability decreases with an increase of temperature and a decrease of the r value [19]. Aluminium hydroxide $Al(OH)_3$ precipitates at pH = 3.0, while at pH above 8.0 (9.0) it starts to dissolve (as an amphoteric compound) [16,20–22].

The optimal pH ranges are: for aluminium sulphate 5.0–7.0 [18] or as reported by other authors 5.0–6.0 [22]; for pre-hydrated polyaluminium chlorides (Flokor, PAX) 4.0–8.0, or as reported by other authors 3.0–9.0 for removal of organic matter [22].

Lower temperatures adversely affect coagulation due to a number of factors such as: lower rates of hydrolysis and hydroxides precipitation, a higher water viscosity (slows down flocs sedimentation), and better stability of colloids [16,23,24]. The presence of aluminium polymeric forms in the coagulant reduces the adverse effect of low temperatures, so pre-hydrolysed coagulants are less sensitive to low temperatures [16].

The grey sewage consists of many organic substances of natural origin with negative charge as well as surfactants – mainly anionic ones and nonionic ones [8,11]. Therefore, positively charged aluminium polymers in a pH environment less than 6 (7) will be good coagulants [18].

The most commonly used surfactants are anionic surfactants and then nonionic surfactants. Cationic surfactants are less frequently used because of their inferior biodegradability. Amphoteric surfactants due to their high cost are used in specialist applications.

Aluminium sulphate and pre-hydrolysed polyaluminium chlorides acidify the coagulated water or wastewater during the hydrolysis reaction, which is advantageous for the formation of positively charged aluminium polymers.

The use of aluminium sulphate or other sulphate coagulants leads to the synthesis of very slightly soluble calcium sulphate (calcium ions are commonly present in natural water). Reuse of such treated sewage in a sanitary installation may contribute to the deposit of calcium sulphate in the installation, thus its contamination or clogging. There will be no such effect in the case of chloride ions derived from poly aluminium chloride. For this reason, the use of pre-hydrolysed poly aluminium chloride will be better in the situation of limited possibilities of retaining micro dispersion suspensions in a small home installation intended for the treatment of gray sewage.

2. Scope

The author searched for a possible low-cost technology for greywater treatment that could be employed in a greywater collection and treatment unit. The treated greywater effluent, after disinfection, could be utilised in agriculture for irrigation or for flushing toilets, car washing and construction works.

The study also focused on developing mathematical models that could assess treatment efficiency for greywater of a similar composition. The models would accommodate the coagulation efficiency and kinetics of photochemical oxidation and predict the overall treatment efficiency for a sequence of processes: coagulation and photochemical oxidation.

The article summarises the results of the coagulation process, only.

3. Materials and methods

3.1. Materials

The research on greywater treatment was based on laboratory scale tests. Greywater came from a small household and included water from: the bathroom (57%), the kitchen sink (29%) and the washing machine (14%). The weekly greywater flow was about 0.28 m^3. The greywater quality parameters varied highly (Table 1), which made it difficult to carry on the study and come up with some general conclusions. By analysing the TOC values, the COD values appear too high. This could be due to the presence of raw non-carbon reducers in the waste water and the conversion of organic carbon into

Table 1
Greywater characteristics

Parameter	Range of values
Permanganate value (PV) [g O_2 /m^3]	96.9–155.0
Chemical oxygen demand (COD) [g O_2 /m^3]	401.3–737.8 (1151.7)
Total organic carbon (TOC) [g C/m^3]	61.52–76.00
Turbidity [NTU]	81.54–143.43
pH	8.48–9.18

inorganic carbon as a result of biological processes occurring in grey sewage collected for several days. There are high values of the COD/TOC quotient (e.g., 7–8 [35]). The greywater was slightly alkaline, mostly due to alkaline soaps. Its temperature was 293 K and all experiments were carried out at this temperature.

3.2. Methods

Permanganate value (PV) and COD have been determined in accordance with the Standard methods [25]. The turbidity measurements were made with the WTW 555IR turbidity meter. Total organic carbon (TOC) was measured with the Formacs HT analyser from Skalar. The Zeta potential was measured with the Zetasiser Nano particle analyser, model ZEN3600.

3.3. Greywater treatment methods

The greywater treatment process comprised several steps. Greywater from the bathroom and the kitchen was pre-filtered with a nylon bag filter with a mesh of 200 µm. The bag filters are produced with mesh openings varying from 5 to over 1,200 µm [26]; a selection of a suitable filter bag has not been a topic of this study. Then, after settling for 0.3 h in a 0.3 m high tank the greywater was coagulated with the pre-hydrolysed poly (aluminium) chloride PAX 16 [27]. The concentrated PAX 16 solution had the following characteristic: pH = 1.0, density = 1.343 g/mL, alkalinity = 36.23% and aluminium concentration = 0.1099 g Al^{3+}/mL.

The greywater was rapidly mixed with coagulant for 1 min at a mixing gradient of 103 s^{-1} and then slowly mixed for 5 min at a mixing gradient of 15 s^{-1}. The precipitated sludge was left to settle for 0.25 h. In small greywater treatment plants (household greywater pretreatment plants) it may be difficult to use coagulation devices for which mixing gradients during flocculation would be on the order of 60 s^{-1}, and flocculation times of 20–40 min. Therefore, the flocculation process was carried out at a low mixing gradient and over a short time period.

After this time, PV, COD and TOC were determined in the settled greywater samples.

3.4. Experimental procedure of coagulation

Preliminary studies have shown that it is possible to effectively treat greywater using doses of coagulant, expressed as dosages D of aluminium of 25–150 g Al^{3+}/m^3. Therefore coagulation of the greywater was carried out with the following doses of coagulant PAX 16: 12.5, 25, 50, 75, 100, 125, 150, 175 and 200 g Al^{3+}/m^3. High coagulant doses may result in high concentrations of residual aluminium. According to the current regulations in Poland [28], the highest acceptable concentration of aluminium in a secondary effluent cannot exceed 3 g Al^{3+}/m^3, in all sectors of the economy.

4. Results and discussion

For coagulant doses expressed as dosages D of aluminium of 25–50 g Al^{3+}/m^3 and for several, independently taken, greywater samples [1,2,3] the lowest residual aluminium concentration was below 1 g Al^{3+}/m^3 (Fig. 1). The doses above 50 Al^{3+}/m^3 may result in residual aluminium concentrations of 1.4 g Al^{3+}/m^3 i.e. still below the highest acceptable value. The study shows that the lowest residual aluminium concentrations do not correspond to zero Zeta potential but rather to potentials varying from –14 to –10 mV (see Figs. 1 and 2).

The lowest effluent turbidity was observed for a coagulant dose range of 25–100 g Al^{3+}/m^3; zeta potential of colloids for these samples varied from –14 to + 14 mV (Fig. 2). A further increase in the coagulant dose may result in a turbidity rise, followed by a turbidity drop (Fig. 2).

Since the coagulant PAX 16 is strongly acid, the pH of the greywater was about 4.3 at the coagulant dose of 200 g Al^{3+}/m^3 (Fig. 3); at the dose of 100 g Al^{3+}/m^3 the pH remained close to 6.

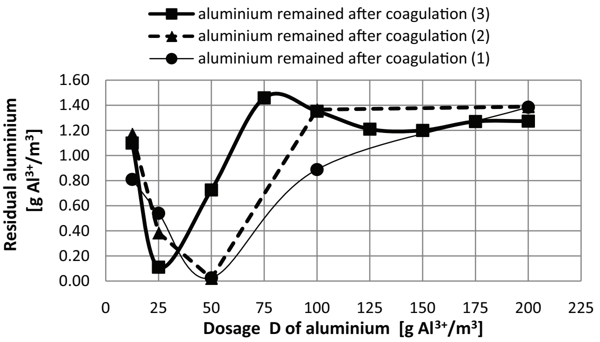

Fig. 1. Aluminium concentrations in greywater after coagulation. (1), (2), (3) – sample number.

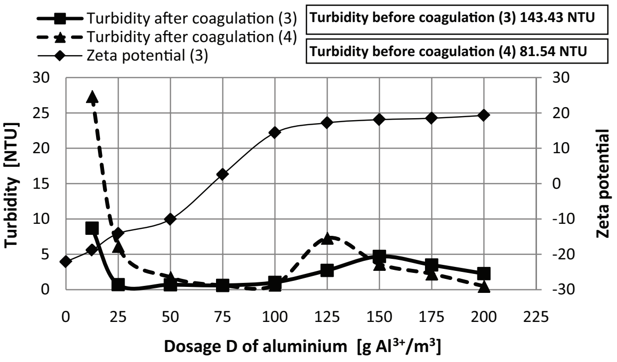

Fig. 2. Changes of turbidity and Zeta potential at different coagulant doses. (3), (4) – sample number.

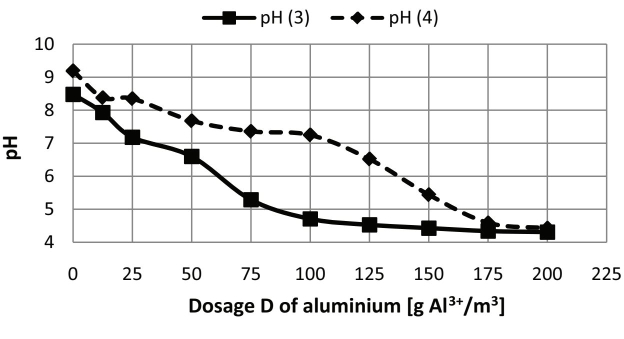

Fig. 3. Changes of pH after dosing the coagulant PAX 16 to greywater. (3), (4) – sample number.

A significant reduction of PV, COD and TOC was observed for coagulant doses up to approximately 100 g Al^{3+}/m^3 (Figs. 2, 4, 6 and 8). A further increase in the coagulant dose was not accompanied by a further reduction of PV, COD, TOC and turbidity; in some cases even a slight increase of these values may be observed (Figs. 2, 4, 6 and 8). Such an effect may be caused by destabilisation of the colloidal system, as a result of adsorption of positively charged aluminium colloids on previously precipitated colloids. The side effect stimulates an increase of the residual aluminium as well as turbidity, PV and COD (Figs. 2, 4, 6 and 8) since the colloids that had already precipitated are transformed into a sol form.

A changing composition of the greywater affects PV, COD, TOC and turbidity removals (Table 2, Figs. 2, 5, 7 and 8). The highest removal was observed for turbidity (about 99%). The removals of other parameters were as follows: PV is ~58%, COD is ~70% (66%) and TOC is ~47% (Table 2).

Within the PV range of 70–90 g O_2/m^3, relatively small changes of COD correspond to relatively large changes of PV; it means that the organic compounds predominantly present in the greywater are easily oxidised by $K_2Cr_2O_7$ and slowly oxidised by $KMnO_4$ (Fig. 9).

In the greywater, a part of organic matter slowly oxidised by $KMnO_4$ but easily oxidised by $K_2Cr_2O_7$ increases with decreasing Al^{3+} doses (below 120 g Al^{3+}/m^3) and therefore the COD/PV ratio increases. Above the dose of 120 g Al^{3+}/m^3, the COD/PV ratio increases again because a share of organic substances slowly oxidised by $KMnO_4$ but easily oxidised by $K_2Cr_2O_7$ and also poorly removed during coagulation increases (Fig. 10).

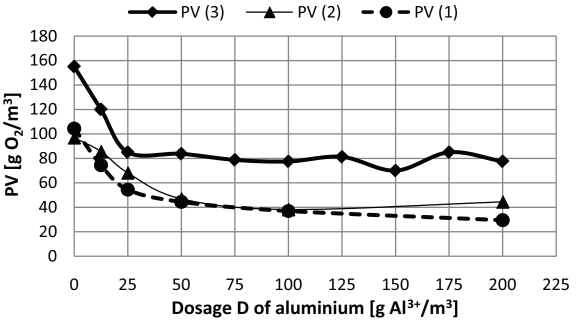

Fig. 4. PV removal vs. coagulant doses. (1), (2), (3) – sample number.

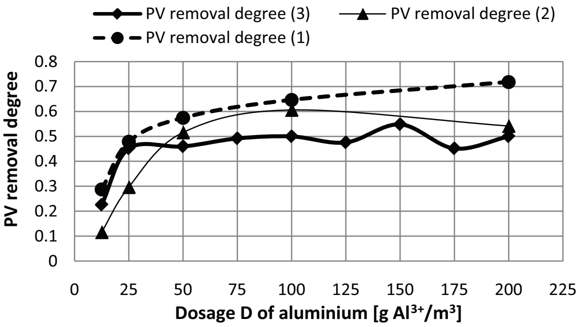

Fig. 5. Efficiency of PV removal vs. coagulant doses. (1), (2), (3) – sample number.

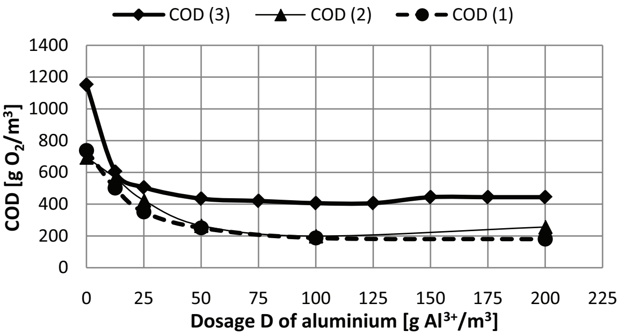

Fig. 6. Removal of COD vs. coagulant doses. (1), (2), (3) – sample number.

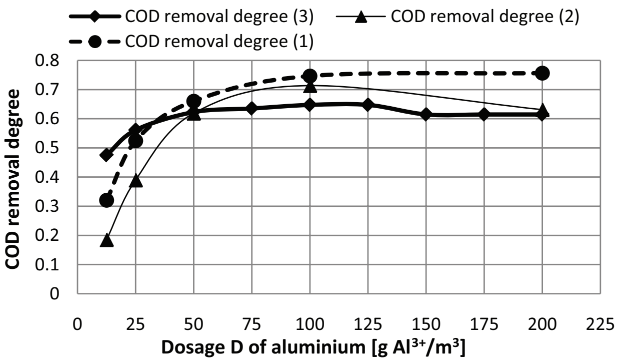

Fig. 7. Efficiency of COD removal vs. coagulant doses. (1), (2), (3) – sample number.

Table 2
Removals of PV, COD, TOC and turbidity at the coagulant dose of 100 g Al^{3+}/m^3

Parameter	Removal (%)
Permanganate value (PV) [g O_2/m^3]	50–65
Chemical oxygen demand (COD) [g O_2/m^3]	65 (56)–75
Total organic carbon (TOC) [g C/m^3]	~47
Turbidity [NTU]	99.2–99.3

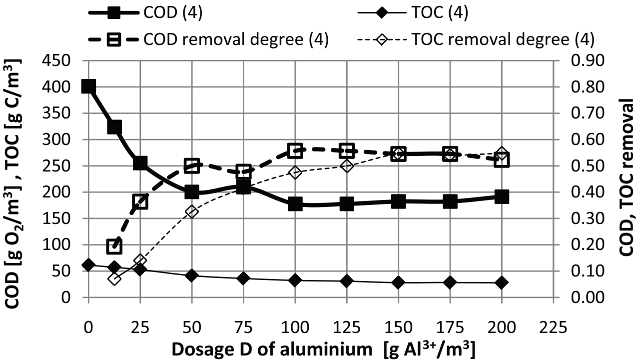

Fig. 8. COD and TOC and their removal efficiencies vs. coagulant doses. (4) – sample number.

4.1. Model of coagulation

A mathematical description of the coagulation effects requires a model presenting the changes in a substance removal per a unit mass of coagulant dC / dD. The differential form of the model is [29]:

$$\frac{dC}{dD} = -k \cdot \Theta^{(T-293)} \cdot C^{a} \cdot D^{b} \tag{1}$$

where C is the concentration of a water quality parameter (PV, COD and TOC); D is the coagulant dose (g Al^{3+}/m^3); k is the proportionality constant –unit corresponds to units of C, D and exponents; a, b are the constants; Θ is the temperature coefficient; T is the temperature of a water sample (K).

Eq. (1) has two solutions:

– if a = 1 then:

$$C = C_0 \exp\left(-\frac{k \cdot \Theta^{(T-293)}}{b'} D^{b'}\right) \tag{2}$$

– if a ≠ 1 then:

$$C = \left(C_0^{a'} - k \cdot \Theta^{(T-293)} \cdot \frac{a'}{b'} D^{b'}\right)^{\frac{1}{a'}} \tag{3}$$

where:

$$a' = 1 - a \tag{4}$$

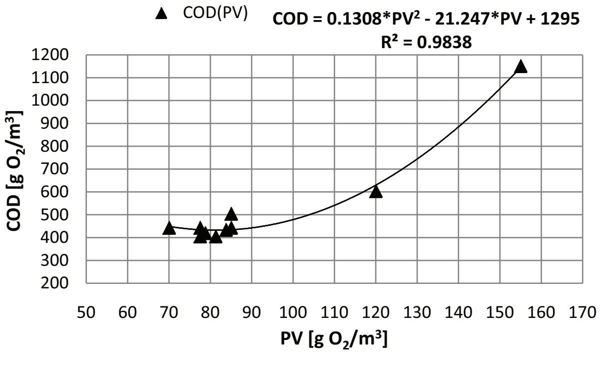

Fig. 9. COD value vs. PV.

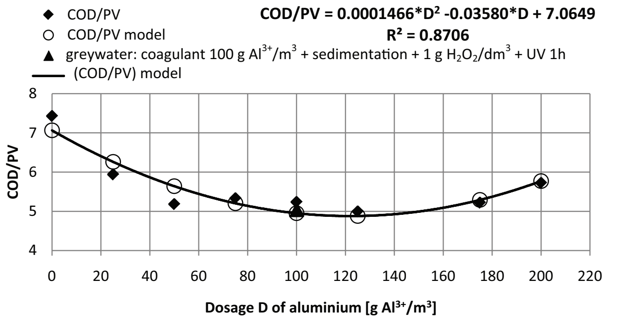

Fig. 10. COD/PV ratio vs. aluminium doses D.

$$b' = 1 + b \tag{5}$$

C_0 is the initial concentrations of water quality parameters (PV, COD and TOC).

Eqs. (2) and (3) make it possible to determine the coagulant dose D once the greywater effluent concentration C (after coagulation) is assumed. Values C_0 and C are enable calculating the coagulation efficiency:

$$\eta = \left(1 - \frac{C}{C_0}\right) \cdot 100\% \tag{6}$$

4.2. Permanganate value removal during coagulation

The PV values obtained after coagulation (T = 293 K) with coagulant PAX 16 are shown in Fig. 11.

The parameters of model (3) were determined by the least squares method applied for the actual PV values and the values calculated from the model. The parameters assumed the following values: k = 3.269, a'= 0.9006 (a = 0.0994), b' = 0.2143 (b = –0.7857).

The average relative error of the model was 0.0983. The fit of the model to the measured data is shown in Fig. 11. Model (3) describes well the PV removal during the coagulation process.

The isolines of efficiency η, presented on the contour plan (Fig. 12) and calculated from Eq. (6) have a positive derivative; it means that an increase of PV corresponds to a reduction of a PV removal, at the same coagulant dose D.

The coefficient a' of model (3) is below 1.0; it means that the coefficient in Eq. (4) is greater than 0. Therefore, an increase of PV will correspond to a higher removal of PV per a unit mass of coagulant (increase of a derivative module dC/dD, Eq. (1)), at a constant coagulant dose D.

The coefficient b' of model (3) is below 1; it means that the coefficients b in Eq. (5) are less than 0. Therefore, an increase of the coagulant dose D will correspond to a lower PV removal per a unit mass of coagulant (decrease of a derivative module dC/dD, Eq. (1)), at a constant PV value. Hence,

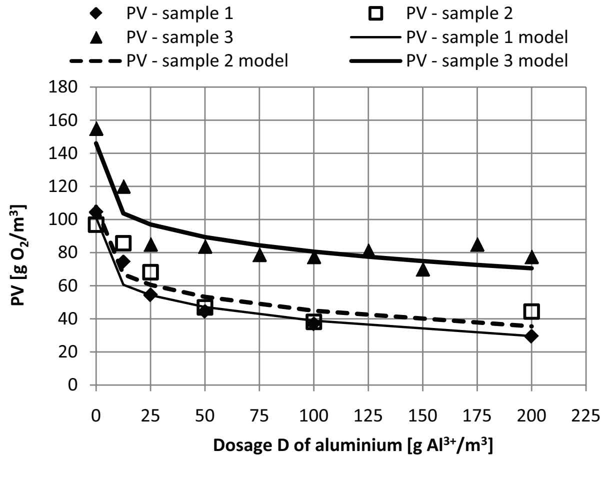

Fig. 11. PV values measured and calculated with the model vs. coagulant doses. 1, 2, 3 – sample number.

higher coagulant doses will result in a lower PV removal per a unit mass of coagulant, i.e. the efficiency of a coagulant usage will be reduced.

4.3. COD removal in a coagulation process

The COD removal during coagulation with coagulant PAX 16 (T = 293 K) is shown in Fig. 13.

The parameters of the model (3) were determined by the least square methods applied to the measured COD and the values calculated from the model. The parameters assumed the following values: k = 0.0482, a'= – 0.0747 (a = 1.0747), b' = 0.2528 (b = –0.7472).

The average relative error of the model was 0.1264. The fit of the model to the measured data is shown in Fig. 13. Model (3) describes well the COD removal during the coagulation process.

The isolines of efficiency η, presented on the contour plan (Fig. 14) and calculated from Eq. (6) have a negative derivative; it means that higher COD corresponds to a slightly higher COD removal, at the same coagulant dose D.

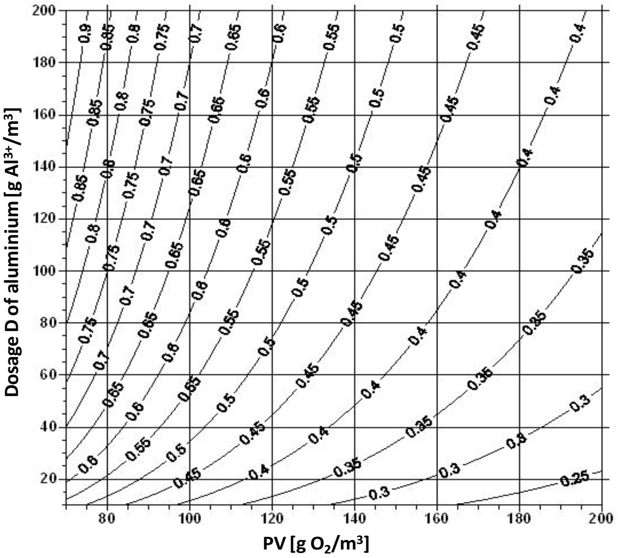

Fig. 12. Contour plan of PV removal efficiencies η.

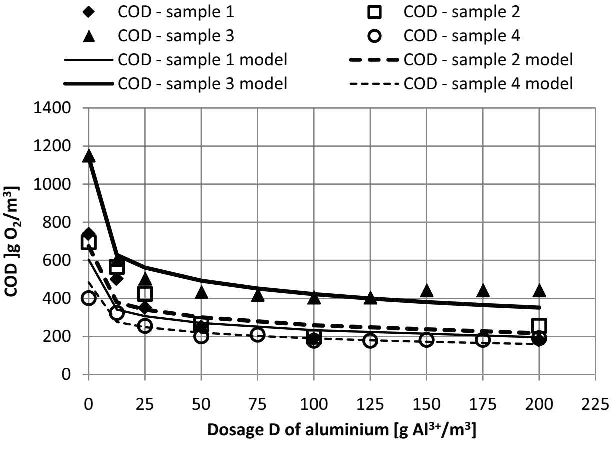

Fig. 13. COD measured and calculated with the model vs. coagulant doses. 1, 2, 3, 4 – sample number.

The coefficient a' of model (3) is below 1.0; it means that the coefficient in Eq. (4) is greater than 0. Therefore, an increase of COD will correspond to a higher amount of COD removed per a unit mass of coagulant (increase of a derivative module dC/dD, Eq. (1)) at the constant coagulant dose D.

The coefficient b' of the model (3) is below 1; it means that the coefficient b, Eq. (5) is less than 0. Therefore, a higher coagulant dose D will correspond to a lower removal of COD per a unit mass of coagulant (decrease of a derivative module dC/dD, Eq.(1)) at the constant COD value. Hence, higher coagulant doses will result in a lower COD removal per a unit mass of coagulant, i.e. the efficiency of a coagulant usage will be reduced.

4.4. TOC removal in coagulation process

The TOC values after coagulation with coagulant PAX 16 (T = 293 K) are shown in Fig. 15.

Parameters of the model (3) were determined by the least squares method applied for the actual TOC values and the

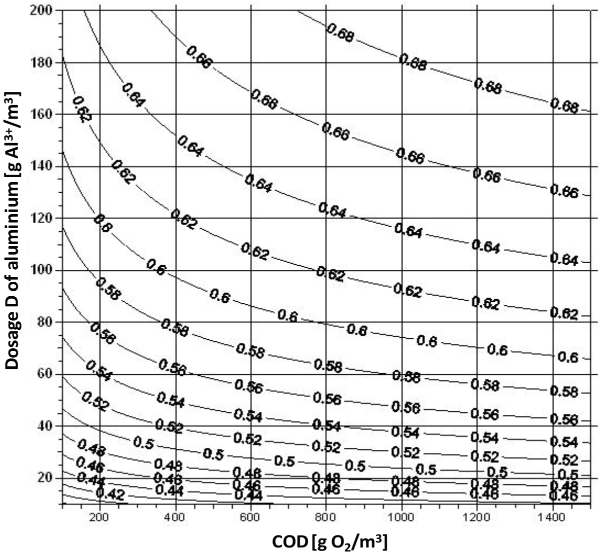

Fig. 14. Contour plan of COD removal efficiencies η.

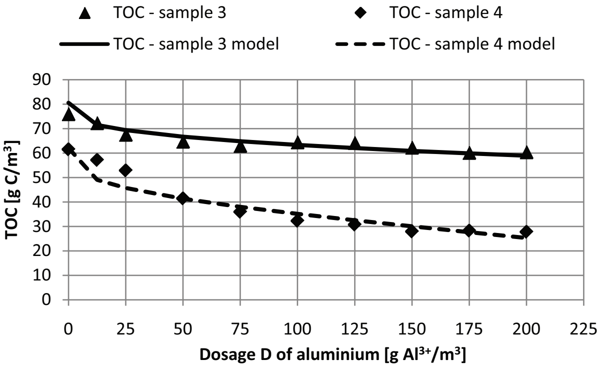

Fig. 15. TOC values measured and calculated with the model vs. coagulant doses. 3, 4 – sample number.

values calculated from the model. The following values of parameters were obtained: $k = 176.17$, $a' = 2.1419$ ($a = -1.1419$), $b' = 0.2776$ ($b = -0.7224$).

The average relative error of the model was 0.0469. The fit of the model to the measured data is shown in Fig. 15. Model (3) describes well the TOC removal during the coagulation process.

The isolines of efficiency η, presented on the contour plan (Fig. 16) and calculated from Eq. (6) have a positive derivative; it means that an increase of TOC corresponds to a lower TOC removal, at the same coagulant dose D.

The coefficient a' of model (3) is above 1.0; it means that the coefficient a in Eq. (4) is below 0. Therefore, an increase of TOC will correspond to a lower removal of TOC per a unit mass of coagulant (decrease of a derivative module dC/dD, Eq. (1)), at the constant coagulant dose D.

The coefficient b' of model (3) is below 1; it means that the coefficients b in Eq. (5) is less than 0. Therefore, an increase of the coagulant dose D will correspond to a lower TOC removal per a unit mass of coagulant (decrease of a derivative module dC/dD, Eq. (1)), at the constant TOC value. Hence, higher coagulant doses will result in a lower TOC removal per a unit mass of coagulant, i.e. the efficiency of a coagulant usage will be reduced.

5. Summary

A coagulation efficiency for raw greywater (COD = 1,200 g O_2/m^3) treated with a coagulant dose of about 100 g Al^{3+}/m^3 was approx. 63% (Fig. 14); the effluent COD was 324 g O_2/m^3. A similar coagulation efficiency (64%) was reported in the studies presented in [30] for the greywater with an initial COD of 791 g O_2/m^3 but at lower aluminium doses (24 g Al^{3+}/m^3). In the work [31] it was reported that for greywater with an initial COD of about 329 g O_2/m^3, COD after coagulation was approx. 152 g O_2/m^3 (54% removal), at a coagulant dose of 34 g Al^{3+}/m^3 and the pH range 4.6–6.4. For high initial COD values (1,200 g O_2/m^3), the coagulant dose must be correspondingly higher, whereas in the dose range of 30–100 g Al^{3+}/m^3 the COD removals vary from 53% to 63% (Fig. 14). The coagulant dose depends on nature of the organic matter present in the greywater, e.g. the surfactants are not always readily removed from the greywater [32–33] or high doses of coagulant are required in proportion to the concentration of the surfactant [34].

6. Principal conclusions

- Polyaluminium chloride based coagulants do not precipitate calcium sulphate (VI). This is important when protecting fittings and an internal sewage installation against the formation of deposits.
- The effective coagulation process is possible at short rapid mixing times (about 1 min) and short flocculation times (about 5 min).
- In the coagulation process, a rapid mixing can be carried out at a mixing gradient of 103 s^{-1} and a slow mixing at a mixing gradient of 15 s^{-1}.
- Short coagulation times and low mixing gradients can be easily implemented at greywater treatment plants.

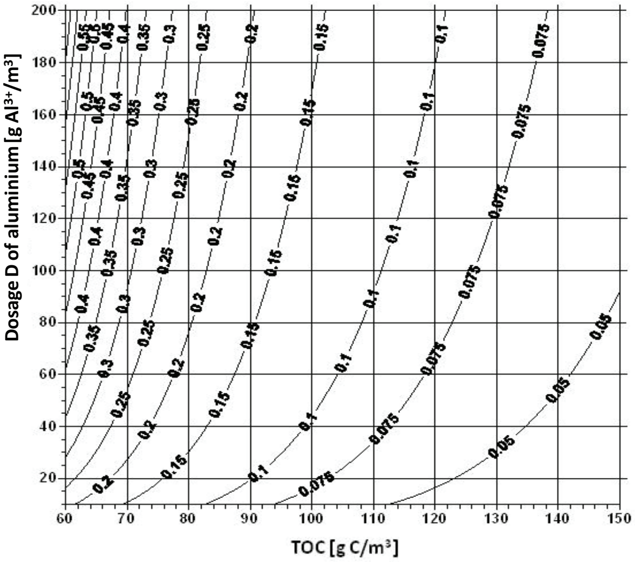

Fig. 16. Contour plan of TOC removal efficiencies η.

- The coagulant PAX 16 can significantly reduce the pH of greywater even to value 4.3.
- Excessive doses of coagulant (above 100 g Al^{3+}/m^3) may result in critical values of Zeta potential, and subsequently in higher greywater turbidity.
- Coagulant doses of 50–200 g Al^{3+}/m^3 may result in the residual aluminium concentration of 1.4 g Al^{3+}/m^3, the value is lower than the one acceptable for the effluent.
- Coefficient a of the coagulation model (1) for COD is 1.0747, therefore, an increase of COD will correspond to a higher COD removal per a unit mass of coagulant (increase of a derivative module dC/dD, Eq. (1)), at the constant coagulant dose D.
- Coefficient b of the coagulation model (1) for COD is –0.7472, therefore, the higher coagulant dose D will correspond to a lower COD removal per a unit mass of coagulant (decrease of a derivative module dC/dD, Eq. (1)), at a constant COD. Hence, higher coagulant doses will result in a lower COD removal per a unit mass of coagulant, i.e. the efficiency of a coagulant usage will be reduced.

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