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1 Nutrient recovery and fractionation of anaerobic digester effluents employing
2 pilot scale membrane technology

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14
15 **Abstract**

16 Anaerobic Digester (AD) waste known as digestate (spent anaerobically digested effluents) of
17 agricultural origin was collected for use in a feasibility study on the use of membrane filtration to
18 fractionate phosphate and ammonia from digestate into nutrient streams. The digestate was pre-
19 treated to remove bulk solids and then filtered using diafiltration (DF) with ultrafiltration (UF) (5.65
20 psi TMP) and then nanofiltration (NF) (operating pressure 253.82 psi). Having set the pre-treated
21 effluents at pH 4.0, retention of phosphate reached 6.78 mmols L⁻¹ during UF with lower values
22 being achieved with repeated DF steps. In contrast, nitrogen retention was lower at 8.21 mmols L⁻¹
23 that were continuously dropping at each DF step. During NF phosphorus was shown to be strongly
24 retained by the membrane at 31.8 mmols L⁻¹, while retention of ammonium was low at 13.4 mmols
25 L⁻¹ demonstrating the potential for this combination of membrane types for fractionating high value
26 components from AD waste.

27
28 *Keywords:* sludge, NF, UF, phosphate, nitrogen, AD

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34

35 **1. Introduction**

36

37 The reduced reliance on naturally occurring carbon sources for energy generation has become a high
38 global priority [1]. The continuously rising cost of fossil fuels as well as the environmental and
39 societal impact of its novel extraction techniques, such as fracking, make the generation of electricity
40 a challenge; therefore, the search for alternative renewable energy sources becomes imperative [2].

41

42 To achieve this goal, several methods of sustainable energy production are explored (e.g. wind, solar
43 tidal *etc.*). To these the combined heat and power option of anaerobic digestion (AD) can be added.
44 AD is an effective and well-established technology for reducing organic waste, stabilising organic
45 materials by conversion to methane, CO₂, NH₃ and other inorganic products [3-5]. It has been used in
46 municipal wastewater treatment; however, it now finds increased application in a range of small and
47 medium sized enterprises (SMEs) where there is a significant quantity of organic waste to deal with.
48 The main advantage of the AD process is the release of carbon as methane gas, but careful
49 consideration must be given to other by products such as NH₃ [6]. When the process of AD comes to
50 an end the resultant viscous liquor is rich in nutrients such as ammonia, phosphate, volatile fatty
51 acids and metals. This creates a waste disposal problem for the operator, since land spreading may
52 be hazardous, causing contamination of the ground and surface waters and leading to
53 eutrophication and concentration in the soil [7, 8].

54

55 However, regardless of the environmental impact [8], these effluents represent a source of valuable
56 chemicals that, if recovered, can be used to further enhance the viability of AD as a means of
57 sustaining the low carbon circular economy [4]. For example, these effluents could be formulated
58 into sterile, large, particle-free fertilising solutions, replacing the highly polluting and expensive
59 production of industrial fertilisers. The commercial production of these fertilisers comprised mainly
60 of ammonia and phosphorus pentoxide are highly polluting as each tonne of ammonia contained
61 generates 2.2 tons of CO₂ to the environment [9], and up to 1.0 tonnes of CO₂ are released per kg of
62 commercial fertilizer [10]. If further fractionated and separated, the nutrients could be of high
63 economic value, since ammonia currently retains a market value of around \$300/ton [11].
64 Phosphate -normally derived from phosphate rock (historically from deposits of guano) and
65 predicted to be depleted within the next 100 years [12]-is used as fertiliser in the form of
66 diammonium phosphate and is currently valued at about \$350/tonne, with the ore itself currently
67 valued at \$100/tonne [13].

68

69 Several methods have been applied to treat AD wastewater or sludge [14], in order to be safely
70 discharged to the open environment. These include biological processes namely bioremediation as
71 well as, energy, time and cost demanding physical (screening, settling, and flotation) and chemical
72 treatments [15]. Commonly these treatments do not allow either the recovery or the reuse of
73 chemicals, leading to the loss of important resources. Numerous other methods have been explored
74 for targeted ammonia and phosphate removal including chemical precipitation within the scope of
75 struvite formation [16] ion exchange and adsorption and ammonia stripping [17]. Contrary to the
76 above-mentioned technologies membrane filtration offers high productivity for relatively low capital
77 and operating costs, as there is no high energy demanding phase changes or addition of solution
78 modifying chemicals. Indeed, it has been reported that in at least ten business areas (desalination,
79 municipal water recycling, industrial process water and waste water treatment, cooling and boiling
80 water treatment and emerging sectors such as oil and gas extraction) the treatment of streams using
81 membranes is expected to see its market value double to 2020 [18,19]. It is easily scalable and can
82 be applied in several arrangements to achieve the desired separation, purification or volume
83 reductions. Previous research [20,21] has shown that membrane filtration has been effectively
84 applied, converting the waste effluent sludge into particle-free nutrient-rich fluid and nutrient-
85 depleted solids stream. Such a strategy leads to a solids fraction with reduced nutrient content being
86 disposed to land as an organic enhancer, while the soluble organic materials, ammonia and
87 phosphate, can be concentrated and formulated into more useful materials and so valorising this
88 route for the wastes. To the authors current knowledge there are limited studies evolving around
89 membrane use for phosphate and ammonia recovery in pilot scale.

90

91 Industrial applications of pressure-driven membrane technology are often accompanied by certain
92 engineering challenges, such as membrane fouling. Fouling is a complex multifactorial phenomenon
93 and is largely but not solely dependent on the feed stream composition [22]. It can be defined as
94 the deposition on the membrane surface of dissolved and undissolved matter forming an
95 undesirable layer causing flux decline. This can occur either due to the deposition of colloidal matter,
96 minerals, and hardness scales. Additionally fouling can be caused by; microbial biomass attachment
97 followed by growth and multiplication due to available nutrients adhered on the membrane surface
98 or in the feed including humic acid and other derivatives of natural organic matter [23]. Fouling is a
99 highly problematic situation, often irreversible, decreasing significantly the separation efficiency of
100 the membranes while increasing production costs due to higher energy demand, additional labour
101 for cleaning and maintenance, use of chemical agents for cleaning and reduction in membrane life

102 expectancy (as fouling reduces the performance of the membranes, regardless of its type). Judicious
103 usage of operating conditions of membrane systems, including temperature and pH, and
104 development of pre-treatment processes such as sedimentation, coagulation, precipitation, dilution,
105 membrane modification and mixing to homogenisation [24-26], wherever possible, can constitute
106 fouling a reversible process and extend the membranes' shelf life. Low-cost, non-chemical pre-
107 treatment, such as dilution, sedimentation, sieving and air flotation are preferable. However,
108 chemical conditioning as a pre-treatment scheme can also be a viable option for systems that
109 require a different method of pre-treatment to meet filtration goals.

110

111 Therefore, the purpose of this study was to ascertain whether it is possible to refine valuable solutes
112 such as nitrogen and phosphate from the waste stream of anaerobic digesters using selected
113 membrane separations in a pilot scale. It thus aims to practically test the applicability of such an
114 operation at a commercialised industrial market, especially considering SMEs, the main AD
115 operators in the UK. The proposal is by using ultrafiltration (UF) and nanofiltration (NF), ammonia
116 and phosphate, can be separated and channelled into enriched streams of reduced overall volume,
117 promoting sustainability and minimising the impact of discharged waste. These streams could be
118 then used effectively as nutrient media used for growing microbes, algae and plants (i.e. hydroponics
119 and aquaponics) with composition tailored to the microorganisms' nutritional needs or as
120 biofertilizers, reducing significantly the cost of production as well as their carbon footprint. Their
121 filterability has been evaluated in terms of flux, membrane resistance and cake resistance, using
122 various operating conditions. Attempts have been made to correlate the solids contents and
123 characteristics with the filterability of sludge using diafiltration treatment scheme. Pretreatment was
124 also investigated to ascertain the effects of acidification and segmentation on nutrient extraction.

125

126 **2. Materials and Methods**

127 **2.1. Materials**

128 Spent anaerobically digested liquid samples (150 L of waste streams of agricultural origin, namely
129 mixed waste of cattle slurry (excretions), vegetable waste (potatoes, apples, carrots and others),
130 maize and grass silage, were taken of the output line of the sedimentation tank before passing
131 through an automatic coarse particle separator (>5mm), from Farm Renewable Environmental
132 Energy Limited (Fre) (<http://www.fre-energy.co.uk/case-studies.htm>), Wrexham, United Kingdom.
133 The spent anaerobically digested effluents have been collected in 25 L plastic jerry cans.

134

135 2.2. Experimental

136 2.2.1. Effluents Pre-Treatment Schemes

137 The effluents were found rich in solids, mostly comprised large particles i.e. straw, stones. Pre-
138 treatment of the raw sludge was required; which was completed by way of acidification, to release
139 phosphate, and then settling. The supernatant fluid was decanted. To ensure undisrupted UF and NF
140 treatment, a pre-treatment scheme was developed to address this problem, combining a set of
141 physical treatments. These include settling, dilution and mixing.

142 In further detail, the samples were left to settle overnight. Physicochemical characterisation of the
143 collected samples (Table 2) demonstrated that spent effluents were rich in solids, mainly coarse
144 particles that could easily block the membrane pores of the UF and NF units. The following day, 50 L
145 of the collected samples were placed in a circular vessel of 0.54 m diameter and 1.3 m height and
146 were diluted in a 1:1 ratio with tap water. Dilution was found helpful in disengaging of the chemicals
147 and nutrients bound in the solids, facilitating their recovery in the permeate. Then thorough mixing,
148 took place, for an hour, with a rod, followed by acidification to pH 4 with HCl 5M. The effluents were
149 then left to settle for 24 h, allowing sedimentation of particles. The supernatant was then collected
150 and filtered by the UF and NF processes. In the case of NF, the supernatant was further treated prior
151 to filtration with a series of coarse filters varying in pore size between 1.045 mm to 0.5 mm.

152 2.2.2. Filtration Unit Design

153 2.2.2.1. Ultrafiltration

154 The waste was processed through a cross-flow UF unit (Fig.1), designed, built and provided by Axium
155 Process, Hendy, Wales, UK. The unit consisted of a 130 L stainless steel vessel (Fig. 1 no. 1) linked via
156 5 m of 1-inch stainless steel piping arranged in two fluid loops each driven by a centrifugal pump,
157 Fristam FPE 722/145B (Fig.1 no 6,14). Waste was passed from the tank into the first pump loop,
158 connected with a pre-filter of 1000 μm (Fig.1 no.7) which pressurised the system against a
159 diaphragm valve (Axium Process, Hendy, Wales, UK) on the return side, which could be adjusted to
160 control the pressure. Within this loop a second pump circuit (centrifugal pump Fristam FPE
161 722/145B) feeding the membrane (KOCH PVDF) (Fig.1 no. 16, 18) enabled high flow rate around the
162 loop. The membrane comprised of 19 channels, of 0.0127 m diameter each and length of 2.921 m,

163 per module (Table 1). The effective membrane area was determined as 4.4 m² (two modules). The
164 membrane was able to withstand a pH range between 2-11, a maximum operating temperature of
165 50°C and had a maximum operating pressure of 87.02 psi. It was fitted in a plastic case commercially
166 available by KOCH (Stafford, UK); while temperature was maintained using a cold water connected
167 cooling heat exchanger provided by Axium Process, Hendy, Wales, UK.

168 There was very little pressure dropping in this loop and thus high fluid velocity over the membrane
169 surface was achieved, which could be kept constant over a range of pressures. All the parts of the
170 unit were connected with stainless steel, heavy duty clamps and sealed with 1.5 inches clamp lipped
171 solid PTFE seals.

172 2.2.2.2. Nanofiltration

173 The pilot scale cross flow nanofiltration unit used for further processing of the waste was designed
174 and fabricated in the Systems and Process Engineering Centre (SPEC), College of Engineering,
175 Swansea University. The unit (Fig.2) was developed operating an industrial standard membrane
176 module within a system that had a limited volumetric retention. The unit consisted of a 25 L stainless
177 steel vessel (Fig.2 no.1) linked via 2.5 m of 3/8 inch stainless steel piping and stainless-steel
178 compression fittings (Swagelok, Bristol, UK) arranged in two fluid loops, each connected to a pump.
179 The first pump was a variable speed, positive-displacement Hydra-cell diaphragm pump
180 (P400NSGSSC050S, Michael Smith Engineers, UK) (Fig.2 no.8); capable of delivering pressures in
181 excess of 652.67 psi. The second pump (M Pumps, T MAG series M2, Michael Smith Engineers, UK)
182 (Fig.2 no.7) was a magnetically coupled peripheral pump operated at fixed speed. This is a low
183 pressure/high flow rate centrifugal pump, essential for providing the desired cross flow velocity in
184 the membrane. Pressure was measured using analogue gauges (Swagelok, Bristol, UK). There was
185 very little pressure dropping across the membrane and as such constant fluid velocity over the
186 membrane surface was achieved. Temperature was measured manually, using a hydrargic
187 thermometer attached in the feed vessel and a coolant coil was incorporated for basic temperature
188 control of the process fluid.

189 The filter employed for this work was a Desal General Electrics DL4040C1025 (Table 1) membrane
190 able to withstand a pH range between 3 and 9 in continuous operation, a maximum operating
191 temperature of 50 °C, maximum operating pressure 600.45 psi (41.4 bar), fitted in stainless steel,
192 commercially available by Lenntech BV (Delft, Netherlands). The membrane has a minimum MgSO₄
193 rejection value of 96% [25, 31]. The effective membrane area was determined as 6.1 m². All the
194 parts of the unit were connected with stainless-steel heavy-duty clamps and sealed with 3/8 inches

195 clamp lipped solid PTFE seals, provided by Swagelok, Bristol UK.

196 2.2.3. Membrane Characterisation

197 2.2.3.1. Ultrafiltration and Nanofiltration

198 Membrane characterisation studies using tap water were carried out to determine the membrane
199 resistance and the influence of pressure during the operation of the systems, UF and NF
200 respectively. The permeability of tap water was measured in order to analyse the behaviour of the
201 system, using a graduated cylinder and a stopwatch. The flux values and cross-flow velocity linearly
202 increased with increasing pressure. For the UF system, water flux increased from 60.90 to 174.37 L
203 $\text{m}^2 \text{h}^{-1}$ with an increase in transmembrane pressure from 5.65 to 20.02 psi, thus cross-flow velocity
204 increased from 2.16 m s^{-1} to 5.44 m s^{-1} . For the NF system the flux increased from 47.35 to 277.92 L
205 $\text{m}^2 \text{h}^{-1}$ with an increase in transmembrane pressure from 5.65 to 20.02 psi, thus cross flow velocity
206 increased from 0.94 m s^{-1} to 5.44 m s^{-1} . The membrane permeability (L) was defined by the slope of
207 the linear functions using the plots of the flux over the TMP. It is a characteristic of the unfouled
208 membrane and was calculated as 7.80 m for the UF system, while the NF system was calculated as
209 8.95 m.

210 2.2.4. Processing Scheme

211 The processing of sludge was carried out using (Fig.3) DF, where the filtration characteristics were
212 studied as a function of dilution of the liquid in the sludge. The purpose of DF was to investigate the
213 effects of removing the soluble components of the sludge. The batch process involved sequential
214 washes which consisted of first concentration and then dilution of the sludge with fresh tap water.
215 Initially for UF, 100 L of the pre-treated sludge was collected and placed in the feed vessel and then
216 concentrated to 50 litres. The permeate was then discarded. In the concentrated sludge, 50 litres in
217 the vessel, 25 L of tap water were added and then processed by the unit, to collect 25 L of permeate.

218 The process was replicated with NF; 30 L of the pre-treated sludge were collected and placed in the
219 feed vessel and then concentrated to 20 litres, the permeate was then discarded. In the
220 concentrated sludge, 10 litres in the vessel, 10 L of tap water were added and then processed by the
221 unit, to collect 10 L of permeate. This was repeated three more times. The permeate flow rate was
222 manually recorded using a graduated vessel, where the permeate fluid was collected. The difference
223 in volume was recorded per minute using a stopwatch (Casio electronics, UK); on a two-decimal
224 points precision electronic scale (OHAUS I-10) (kilograms, kg).

225

226 **2.2.5. Analysis of dry matter content and physicochemical characteristics**

227 Total solids (TS, g L⁻¹), total suspended solids (TSS, mg L⁻¹), total dissolved solids (TDS, mg L⁻¹),
 228 alkalinity, and optical density were determined according to APHA, 1998. Nitrogen was measured as
 229 ammonia (NH₃-N) using the phenate colorimetric method, where ammonia reacts with phenol to
 230 form indophenol complex in the presence of alkali and an oxidizing agent. Sodium nitroprusside acts
 231 as catalyst and the developed blue color absorbs light at 640 nm wavelength. Phosphorous (PO₄-P)
 232 was measured using vanadomolybdo-phosphoric acid colorimetric methods as described by APHA,
 233 1998 at 470 nm. A spectrophotometer UV-Visible UNICAM UV300 dual beam was used for both
 234 methods. Each parameter was triplicated to obtain the average data (standard deviation of mean
 235 <5%, standard error <7%) offering highly significant results. When necessary, samples were diluted
 236 with deionized water to fit within the calibration range. Particle size distribution (PSD) of the sludge
 237 samples was determined by light scattering technique using Mastersizer 2000 (Malvern, UK), the
 238 zeta potential was determined by the Zetasizer (Malvern, UK), the conductivity and salinity of the
 239 samples were measured using a conductivity meter (Russell systems, UK) calibrated with a standard
 240 solution of 0.1M of KCl.

241 **2.3. Theoretical**242 **2.3.1. Determination of the Filtration Parameters**

243 For the determination of flux and other parameters the following equations [25-27] were used

244

245 Permeate flux (permeate)

$$246 \quad J_{permeate} = \left(\frac{Q_f}{A_m} \right) = \left(\frac{dV}{dt} \right) \quad [1]$$

247

248

249 Flux (J) in the system was determined as

250

$$251 \quad J = \left[\frac{\Delta P - \Pi}{(R_m + R_c) * \mu} \right] \quad [2]$$

252

253

254
255 Transmembrane pressure (ΔP) was defined as

256
257
$$\Delta P = \text{TMP} = \left(\frac{P_{\text{inl}} + P_{\text{out}}}{2} \right) - P_{\text{permeate}}$$

258 [3]

259 The total membrane resistance [25-27] was also calculated by

260
$$R_T = (R_m + R_c)$$

261 [4]

262 where the membrane resistance was defined by Darcy's law [26-29] as

263
$$R_m = \frac{\Delta P}{J * \mu}$$

264 [5]

265 that for the calculation of the cake resistance [26-29] becomes

266
$$R_c = \left(\frac{\Delta P}{J * \mu} \right) - R_m$$

267 [6]

268 where the R_m equals to the R_m of water under the same operating conditions.

269 Cross flow velocity was defined as following

270
$$U = \frac{Q_f}{\pi * r^2 * n}$$

271

272

273

274 3. Results and Discussion

275 3.1. Physical Characteristics of Agricultural Waste Effluent Streams

276 One hundred and fifty liters (150 L) sludge samples were taken from the anaerobic digester without
277 any on site processing. These materials required some pretreatment to allow the sludge to be easily
278 handled within the filtration unit. As the collected sludge was considered high in content of
279 suspended solids, gravity based primary treatment was applied. This enhanced the removal of larger
280 particulates of the anaerobically digested effluents ($<100\ \mu\text{m}$) and facilitated their filterability
281 through the polysulfone filter. The spend anaerobically digested effluents were placed in a
282 circulatory tank of 0.54 m and height of 1.5m and diluted by 50% v/v with tap water. It has been
283 found from previously published work that phosphate molecules are loosely bound on the solids
284 surface [21,29] therefore dilution's scope is to move phosphate ions in the supernatant. After
285 thorough continuous mixing for at least an hour with a wooden rod, the effluents are left to settle
286 for 24h. The supernatant is collected from the top of the settling vessel and used in the studies of
287 ultrafiltration and nanofiltration.

288 Reduction to the total solids content by 44.4% ($55.42\ \text{g L}^{-1}$ to $30.81\ \text{g L}^{-1}$) was observed; in total
289 dissolved solids a reduction of 60% was observed ($31107.8\ \text{mg L}^{-1}$ to $12443.12\ \text{mg L}^{-1}$) in color by 15
290 % (0.18 to 0.153 at 580nm). Significant reduction was observed in the TSS content, 46.70 %, thus
291 making the effluent to be filtered a simpler material to be processed (Table 2).

292 However, in addition to the successful removal of large particulate matter, it was found that in terms
293 of nutrients ammonia and alkalinity (defined as equivalent to $\text{CaCO}_3\ \text{mg L}^{-1}$) were reduced while the
294 scheme had a limited effect on conductivity and size. These successfully recovered materials of
295 interest can be formulated, through further processing with membrane technology i.e. UF and NF
296 into effluents suitable for use as biofertilizers or as nutrient media for microbial fermentations, so to
297 produce biofuels and chemicals.

298

299

300

301

302 3.3. Filtration Characteristics of Anaerobically Digested Effluents using 303 Diafiltration Strategy

304 3.3.1. Ultrafiltration

305 The effluents were filtered in the dual loop UF system, using diafiltration (Fig.3) under constant
306 temperature and pressure control, with one centrifugal pump being used in a recirculation loop to
307 maintain high constant fluid velocity across the membrane while the second pump introduced the
308 fluid and pressurized the system, establishing a cross-flow UF system.

309 The filterability of the digested effluents was evaluated in terms of flux, total membrane resistance
310 and cake resistance. At 5.65 psi TMP (Table 3), flux (eq. 1, section 2.3.1.) varied between 268.9
311 $261.7 \text{ L m}^2 \text{ h}^{-1}$ Over the course of the filtration, the total membrane resistance gradually increased,
312 $1.86 \cdot 10^{13}$ to $2.53 \cdot 10^{13} \text{ m}^{-1}$, due to the continuous deposition of matter on the membrane channels,
313 since particulates larger than the membranes pore size are retained. A cake was formed on the inner
314 surface of the membrane channels, reflected by the development of the cake resistance at each
315 washing step, varying between $3.28 \cdot 10^{11}$ and $4.78 \cdot 10^{11} \text{ m}^{-1}$. The leaching process has an effect on
316 the composition of the digested fluids in the feed, with a mean size drop of particulates from 17.73
317 μm to $13.99 \mu\text{m}$. This is further reflected by the decreased amount of particles in the feed at each
318 step of the process with TS from 30.60 to 17.47 g L^{-1} , TSS varying between 547.90 mg L^{-1} to 237.90
319 mg L^{-1} and TDS from $8482.40 \text{ mg L}^{-1}$ to $3425.25 \text{ mg L}^{-1}$, a total reduction of 59.62% (Table 3).
320 Consequently, the effect of the cake resistance is minimized; the fluids are transferred across the
321 membrane, leaving the flux relatively unaffected. The cake is presumably permeable due to the
322 diafiltration pattern followed that allows its continuous leaching, altering significantly the chemical
323 properties of the digested effluents. The changing content of ions, due to the hydrolysis of the ionic
324 bonds is shown by the gradual reduction of conductivity (9.98 mS cm^{-1} to 4.03 mS cm^{-1}) and alkalinity
325 ($3750 \text{ mg CaCO}_3 \text{ L}^{-1}$ to $1250 \text{ mg CaCO}_3 \text{ L}^{-1}$) and positively influences the filterability of the digested
326 fluids. This is done by consisting the particles less absorbent to the membrane surface, reducing the
327 particulate- membrane interactions, therefore reducing electro-viscous effects, allowing the
328 continuous filtration of streams in low pressure operation.

329 This benefit greatly the operation of the system into the present length of operation , since
330 interruptions due to cleaning of the system with expensive chemical agents or back flushing are
331 avoided. However, zeta potential remains elevated possibly due to the existence of several other
332 charged particulates in the mixture. The color of the digested effluents was successfully removed

333 (OD_{580nm} from 0.087 to 0.016) through the three leaching stages of this process (Table
334 3). Consequently the process treats effectively the organic matter content in the digested effluents,
335 since color is commonly caused by organic decomposition products from vegetation or a result of
336 impurities of minerals such as iron and manganese.

337 3.3.2. Nanofiltration

338 The effluents that were filtered in the NF system were previously filtered using the UF equipment
339 (dewatering step) (Fig.3). The streams were filtered under constant operating pressure of 253.82 psi,
340 temperature and pressure control. The filterability of the effluents and the overall behavior of the
341 unit were investigated using the parameters of total membrane resistance and flux. Flux remained
342 relatively constant across the filtration process from 152.6 to 156.6 L m² h⁻¹ while membrane
343 resistance did not vary significantly during the diafiltration process, 2.06*10¹³ to 3.16*10¹³ m⁻¹
344 (Table 4). The slight variations in membrane resistance are indicative of a solids deposition across
345 the filter, however this phenomenon does not seem to influence the flux. On the other hand, a
346 reduction is being observed during the process in TDS (7020.5 mg L⁻¹ to 3688.73 mg L⁻¹, 47.45%
347 reduction), TSS (190 mg L⁻¹ to 70.60 mg L⁻¹) optical density (0.0648 to 0.0356) and sizing (from 16.32
348 μm to 9.69μm).

349 The ionic content of the effluents remained almost unaffected during the leaching steps (Table 4),
350 apart from an initial drop at the dewatering step due to the retention of particulate matter from the
351 membrane (conductivity 4.18 mS cm⁻¹ to 4.34 mS cm⁻¹, alkalinity 3500 mg CaCO₃ L⁻¹ to 3125 mg
352 CaCO₃ L⁻¹, pH 7.65 to 7.32, and zeta potential -33.30 mV to -31.80 mV) making NF an ideal candidate
353 for formulation of effluents. Diafiltration does not seem to have such a strong effect on the physical
354 characteristics of the feed, including solids content contrary to the case of UF where the added
355 water continuously washes the loosely attached particulate matter on the membrane surface
356 breaking the ionic bonds and changing significantly the content in the solutions. In the case of
357 nanofiltration, diafiltration is serving as an aid, facilitating the flux and avoiding disruptions due to
358 membrane pore swelling or pore blockage since the system is operated in continuous mode.

359 3.4. Nutrient Extraction using Ultrafiltration

360 The pH of the raw material was adjusted to 4 during the pretreatment stage such that phosphate
361 may be in solution as phosphate ions, since previous work [28] has shown this being an effective
362 measure releasing at least 5% more phosphate in the permeate with no influence on the recovery of
363 nitrogen. Relevant to the nature of UF and more significantly of NF membranes, the chemical

364 speciation of phosphorus and ammonia, is essential for comprehending their separation of one to
365 another. The pH of the waste stream, in this study has been determined as 4, imposes the speciation
366 of each solute (Fig.4), that for ammonia would be NH_4^+ and for phosphate would be $\text{H}_3\text{PO}_4/\text{H}_2\text{PO}_4^-$
367 [29]. The original concentration of phosphate was 9.5 mmol L^{-1} and ammonia was $47.21 \text{ mmol L}^{-1}$.
368 The UF process separated any suspended particles, pathogens and colloidal agents above 500 kDa
369 thus preparing the nutrient-rich solution for the subsequent NF process. Such recovery process is
370 vital for effective nutrient fractionation using NF membranes. Ammonia was found to reach the
371 permeate as a significant proportion of that measured in the feed (Fig.5). With consecutive DF steps
372 a large amount of ammonia is reaching the permeate, emphasizing the importance of DF, as a
373 recovery technique, since ammonia is continuously washed off the compressible permeable cake
374 formed on the membrane surface.

375 During each DF step of UF and NF processes, a permeate and a retentate sample were collected
376 separately at and analysed for nitrogen as NH_3 and phosphorus as PO_4 . In the first concentration
377 step of UF, phosphate concentration in P1 (permeate 1) was very low (Fig.5). Phosphate was
378 retained in the feed side (retentates, Fig.6), while no concentration effect was being found. Initial
379 ammonium concentration was found to be $47.21 \text{ mmol L}^{-1}$. The overall trend in the data is towards
380 ammonium depletion, namely the rejection of ammonia to the permeate, as expected, since
381 ammonium would not be subject to high retention levels at a UF membrane (Fig.6).

382 Furthermore, concentration effect on the ammonium in the retentate (feed side) was not observed
383 (Fig.6). On the feed side, ammonium concentration decreased during the concentration step (R1),
384 suggesting that a negative rejection effect was occurring, which consequently led to higher
385 concentrations of ammonium in the permeate (Fig.5). This negative rejection could be due the
386 presence of an additional chemical species (other charged ions) in the anaerobically digested spend
387 samples that enhanced the transport of NH_4^+ across the filter. Whatever the nature of the process, it
388 could be seen that the DF process was producing the desired result of formulation of separation
389 between phosphate and ammonium, resulting ammonium rich/phosphate limited permeate
390 solutions.

391 Analysis of total solids content for this run revealed that the transport of total solids across the
392 membrane was unimpeded even though the solids content value commenced from similar order of
393 magnitude. This suggests that most of the solids are present as small dissolved species and not
394 larger suspended matter. This theory is supported by inspection of the total suspended solids data
395 that shows the TSS to be of much lower concentration (Table 3).

396 3.5. Nutrient Extraction using Nanofiltration

397 As for the UF stage, four diafiltration steps were completed during the second stage NF treatment
398 for nutrient recovery. The initial concentration of phosphate was 18.8 mmol L^{-1} and ammonia was
399 $45.32 \text{ mmol L}^{-1}$. The aim of this stage of membrane filtration was to separate the phosphate and
400 ammonium into two enriched streams (Fig.4). To achieve this the Osmonics DL membrane was
401 selected, as it is described as having a salt rejection of 96%, a low molecular weight cut-off (Table 1)
402 and in a preceding study this membrane performed well during bench trials [30,31]. Ideally retention
403 at the DL membrane should be high for phosphate and low for ammonium.

404 In order to achieve the desired separation of phosphate and ammonium, the transport of
405 ammonium through the DL membrane would need to be high and that of phosphate low.
406 Theoretically this should be the case, since the ammonium ion is small, it has a molecular mass
407 identical to water and it carries a positive charge which will facilitate its transport towards a typically
408 negatively charged membrane. The data from this trial supports this assumption, since it is apparent
409 that the majority of ammonium ended up in the permeate and the feed was depleted of ammonium,
410 decreasing from a total value of $45.32 \text{ mmol L}^{-1}$ (in 13L of feed) in the initial feed to 3.6 mmol L^{-1} in
411 the final retentate (3L), a reduction of 92.05%.

412 At the beginning of the NF stage, ammonium was present at 2.4 times the concentration of
413 phosphate. However, by DF step 4 the ammonium was present only as a very small fraction of the
414 prevalent phosphate concentration (Fig.8) in the permeate. The aim of the NF stage using a suitable
415 membrane was to separate ammonium and phosphate nutrient ions, since ammonium is a very
416 small molecule. In practice phosphate was determined to be well retained by the DL membrane,
417 whilst a significant proportion of the initial ammonium load (93%) was transported through the
418 membrane to the permeate.

419 This purification step demonstrates the possibility to formulate solutions of nitrogen with virtually
420 no phosphorus present in solution. Nevertheless, the continuous retention of phosphorus increased
421 the concentration of phosphorus in the retentate with residual amounts of phosphorus still present.
422 The drawback of this procedure was the increasingly diluted permeate stream which resulted using
423 DF. However, the recovery and fractionation of nutrients from waste sludge is a vital step in the
424 valorization of wastewater and waste sludge. In particular, dairy manure digestate contain generous
425 quantities of nutrients, up to $3000 \text{ mg L}^{-1} \text{ NH}_3\text{-N}$ have been reported for dairy manure digestate [32]
426 that could be further separated using membrane filtration systems. Filtration treatment of waste
427 effluent for size reduction and decontamination has been proposed in the literature and applied in

428 the industry [33,34]. The pre-treatment scheme had effectively removed a large part of the solids
429 due to the effluents were filtered through a cross filtration unit equipped with an ultrafiltration
430 membrane. When DF is applied, cake resistance was considerably reduced during ultrafiltration. At
431 the final sequential step, the highest cake resistance occurred (Table 3), due to the formation of a
432 compressible permeable cake. The flux remains elevated throughout the process despite of the
433 retention of particles by the membrane, therefore the cake is permeable, allowing the continuous
434 operation of the system in lower transmembrane pressures.

435 There is a dependence of the system on the TS, since the cake resistance increased (Table 3)
436 resulting in lower flux and consequently lower productivity. The cake resistance can be correlated
437 also with the size of the solids and the ionic properties of the digested fluids reflected by the zeta
438 potential. In this case, DF is proven beneficial and -effective; treating the commonly faced problem
439 of formation of insoluble salts deposits on the membrane surface.

440 This treatment can possibly ensure the formulation of microbial and particle free effluents, safe for
441 disposal in the landfills. Animal waste can cause health hazards related to microbial load as well as
442 toxic compounds that can be potentially dangerous to human health. Membrane filtration offers a
443 viable alternative to the current techniques for waste management.

444 Having, therefore, successfully valorized the effluents by removing coarse particles, indigenous
445 microbial/viral load, toxic substances and colorants, the produced effluents can be used as source of
446 nutrients, organics and salts that when precisely formulated, can serve as fertilizer and growth
447 medium for microbial production of platform chemicals and biofuels. Filtration allows manipulation
448 of the nutrient content, since it can be combined with leaching and acidification using UF, for
449 selective separation and concentration using subsequent NF. Within this context, when DF is
450 applied, effluents are produced in different ratios of nutrients content. Each washing step reduces
451 the amount of nutrients in the effluents, gradually depleting the digested sludge and making it safe
452 for disposal in the environment. The depleted sludge, if found containing an amount of phosphate
453 and ammonia can be recycled by being placed back in the processing system. The processing time
454 needed for each step is low (Fig. 6,8), the operation of the system -due to elevated flux and cross
455 flow velocity- make DF a highly effective system in terms of productivity and fluids processability.

456 These effluents, if used as nutrient media [35, 36], are potentially highly profitable, especially when
457 compared to the traditional synthetic media or that derived from food sources such as crops. The
458 composition of these effluents can be modified accordingly to address specific nutritional needs of
459 industrially relevant microorganisms. In terms of nutrient production, the concentration of

460 substances of interest in the effluents remains constant, allowing limited manipulation and
461 benefiting only in volume reduction and nutrient depletion

462 This approach has also other advantages including the use of recycled materials instead of newly
463 synthesized or mined materials; the reduction in the volume and concentration of waste resulting in
464 reduction of demand and costs in waste treatment plants; the creation of valuable streams such as
465 nutrient streams for application in agriculture and bioprocessing.

466 4. Conclusions

467 These results suggest that complex effluent streams such as spent anaerobic digester effluents -after
468 pre-treatment and screening to remove the large particles- can be filtered and fractionated with a
469 series of crossflow filtration UF and NF filters.

470 • The pre-treatment scheme applied achieved a reduction of the total solids of 44.4% (55.42 g L⁻¹ to
471 30.81 g L⁻¹); in total dissolved solids a reduction of 60% was observed (31107.8 mg L⁻¹ to 12443.12
472 mg L⁻¹) and in color 51.66 % (0.18 to 0.153 at 580nm).

473 • Digested agricultural sludge can be effectively filtered through a tubular ultrafiltration unit after
474 pre-treatment at a 268.9 L m² h⁻¹

475 •DF contributes to the independence of the flux rates to the cake resistance; this is explained by the
476 formation of a compressible permeable cake layer that allows the continuous operation of the
477 ultrafiltration system, under constant low-pressure condition (TMP 15 psi).

478 • NF effectively fractionates the effluents into nutrient rich streams of varying concentration of
479 phosphate and ammonia.

480 Membrane processing can possibly become a viable alternative to the development of nutrient-rich,
481 particle-free waste-based solutions, which could have numerous profitable applications, such as
482 fertilizers or specifically tailored nutrient media.

483

484

485

Nomenclature

U	Cross flow velocity	m s^{-1}
Q_f	Volumetric flow rate	L h^{-1}
π	Mathematical constant (3.14159)	
r	Radius	m
n	Number of membrane channels	
J_{permeate}	Permeate flux	$\text{L m}^2 \text{ h}^{-1}$
A_m	Cross-sectional area	m^2
$\frac{dV}{dt}$	Volumetric flow rate (L h^{-1}) where dV: volume differential (L); dt: time differential (min)	
J	Flux	$\text{L m}^2 \text{ h}^{-1}$
ΔP	Pressure differential	psi
Π	Osmotic pressure	psi
R_m	Membrane resistance	m^{-1}
R_c	Cake resistance	m^{-1}
μ	Viscosity (water)	$\text{N m}^2 \text{ s}^{-1}$
TMP	Transmembrane pressure	psi
P_{in}	Pressure inlet	psi
P_{out}	Pressure outlet	psi
P_{permeate}	Pressure permeate	psi
R_t	Total membrane resistance	m^{-1}

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Characteristics	Membranes	
	Ultrafiltration	Nanofiltration
Manufacturer	KOCH	General Electric -Osmo
Model	Super-Cor HFM-513	DL
Distributors	KOCH	Sterlitech Corporation http://www.sterlitech
Material	Polyvinylidene difluoride (PVDF)	Thin film composite polyamide microporous
Applications	Juice Processing	Water Softening, Acid Detergent removal, He
Geometry	Tubular	Spiral wound
Effective Membrane area (m²)	4.4	6.1
Flux rate (L m² h⁻¹ at 99.93 psi)	-	52.7
Charge (at neutral pH)	Neutral	Negative
pH	2-10	3-9
Ion rejection (%)	-	96
MWCO (Da)	500,000	150-300
Contact angle (Θ°)	-	51
Maximum Operating Temperature (°C)	49	50
Maximum Operating Pressure (bar)	6.2	41.4

Table 1: Membranes characteristics provided by the manufacturers and in the literature [25,31]

Physicochemical parameters	Untreated sludge	Pre-treated Sludge
Total Solids (TS, g L⁻¹)	55.42	30.81
Total Suspended Solids (TSS, mg L⁻¹)	1369.75	730.00
Total Dissolved Solids (TDS, mg L⁻¹)	31107.8	12443.12
Conductivity (mS cm⁻¹)	18.67	14.64
Alkalinity (mg CaCO₃ L⁻¹)	8125	4125
Optical Density (580 nm)¹	0.18	0.153
pH	8.9	8.5
Zeta potential (mV)	-38.50	-36.90
Mean Particle Size (μm)	29.51	19.90

Table 2: The physicochemical characteristics of the anaerobically **digested agricultural sludge**

Physicochemical Parameters (Diafiltration Strategy) ²	Water	Dewatering Step	Washing step 1	Washing step 2
Flux (J, L m ² h ⁻¹)	1076	268.9	214.9	215.3
Total Membrane Resistance (R _t , m ⁻¹)	2.88*10 ¹²	1.86*10 ¹³	1.94*10 ¹³	1.90*10 ¹³
Cake Resistance (R _c , m ⁻¹)	-	3.28*10 ¹¹	6.73*10 ¹¹	5.00*10 ¹¹
Total Solids (TS, g L ⁻¹)	-	30.60	22.17	17.72
Total Suspended Solids (TSS, mg L ⁻¹)	-	547.90	321.60	253.20
Total Dissolved Solids (TDS, mg L ⁻¹)	-	8482.40	8133.5	5541.60
Conductivity (mS cm ⁻¹)	0.004	9.98	9.51	6.52
Alkalinity (mg CaCO ₃ L ⁻¹)	-	3750	2500	1875
Optical Density (580 nm) ³	0.00	0.087	0.093	0.015
pH	6.5	7.97	7.88	7.91
Zeta potential (mV)	-2.3	-35.70	-33.52	-32.02
Mean Particle Size (µm)	0.00206	18.95	17.73	14.43

Table 3: Changes in flux and membrane resistance, physical and chemical characteristics of digested agricultural sludge in ultrafiltration membrane

² The filtration characteristics were studied a function of the concentration and dilution of pretreated microfiltered sludge as described in the text.
³ The collected samples were diluted 100 times with deionised water and measured in a 1 cm light path

Physicochemical Parameters (Diafiltration Strategy)⁴	Water	Treated UF Sludge	Dewatering Step	Washing step 1	Washing
Flux (J, L m² h⁻¹)	730.8	-	152.6	129.2	146.9
Total Membrane Resistance (R_t, m⁻¹)	2.88*10 ¹²	-	2.06*10 ¹³	9.69*10 ¹²	1.40*10 ¹³
Total Solids (TS, g L⁻¹)	-	23.64	20.05	17.78	17.25
Total Suspended Solids (TSS, mg L⁻¹)	-	190.1	106.5	149.15	78.10
Total Dissolved Solids (TDS, mg L⁻¹)	-	7020.50	5414.11	3552.74	3620.74
Conductivity (mS cm⁻¹)	0.004	8.26	6.37	4.18	4.16
Alkalinity (mg CaCO₃ L⁻¹)	-	5000	3750	3500	3250
Optical Density (580 nm)⁵	0.00	0.0648	0.0524	0.0159	0.0724
pH	6.5	8.26	7.65	7.55	7.35
Zeta potential (mV)	-2.3	-34.05	-33.52	-33.30	-32.02
Mean Particle Size (μm)	0.00206	16.32	16.04	11.58	10.36

Table 4: Changes in flux and membrane resistance, physical and chemical characteristics of **digested agricultural sludge** in nanofiltration membrane

⁴ The filtration characteristics were studied a function of the concentration and dilution of pretreated microfiltered sludge as described in the text.

⁵ The collected samples were diluted 100 times with deionised water and measured in a 1 cm light path

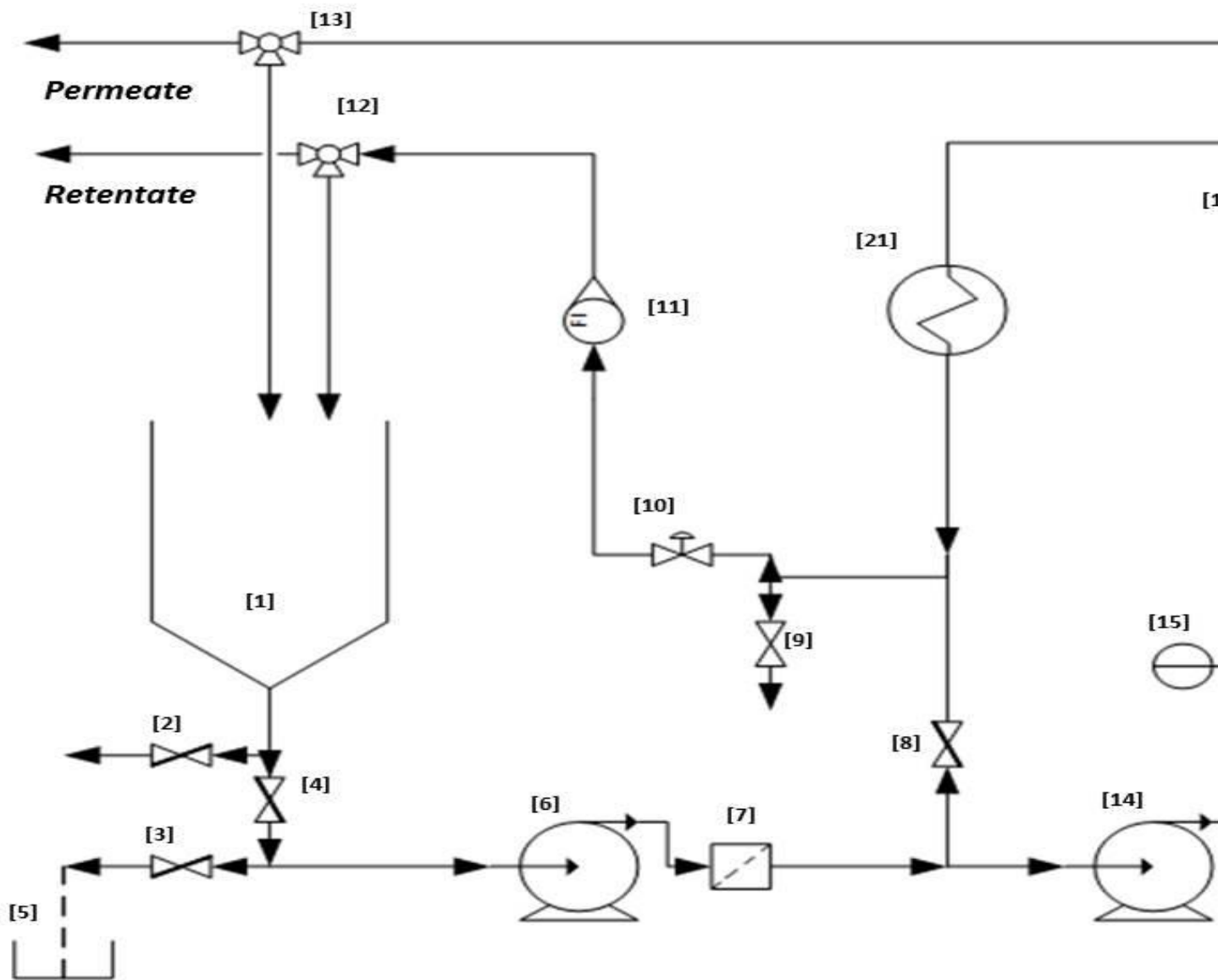


Fig. 1. Schematic diagram of pilot scale ultrafiltration unit : [1] feed vessel (130 L), [2,3,4] butterfly valve, [5] drain, [6] feed pump, [9] sample port, [10] diaphragm valve, [11] rotameter, [12,13] three way valve, [14] regenerative pump, [15] pressure gauge, temperature gauge, [18] ultrafiltration membrane, [19] pressure gauge, [20] rotameter, [21] heat exchanger

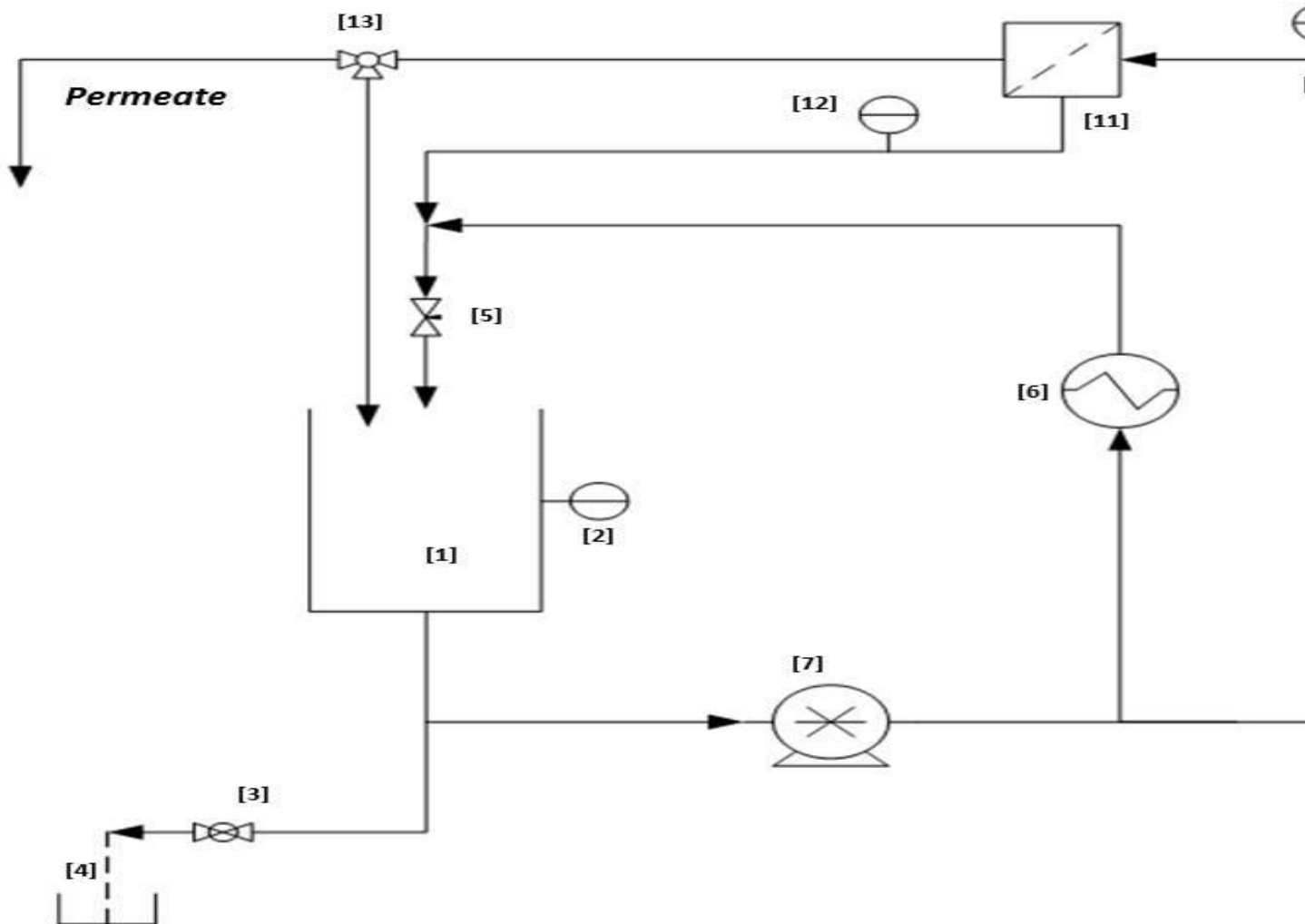
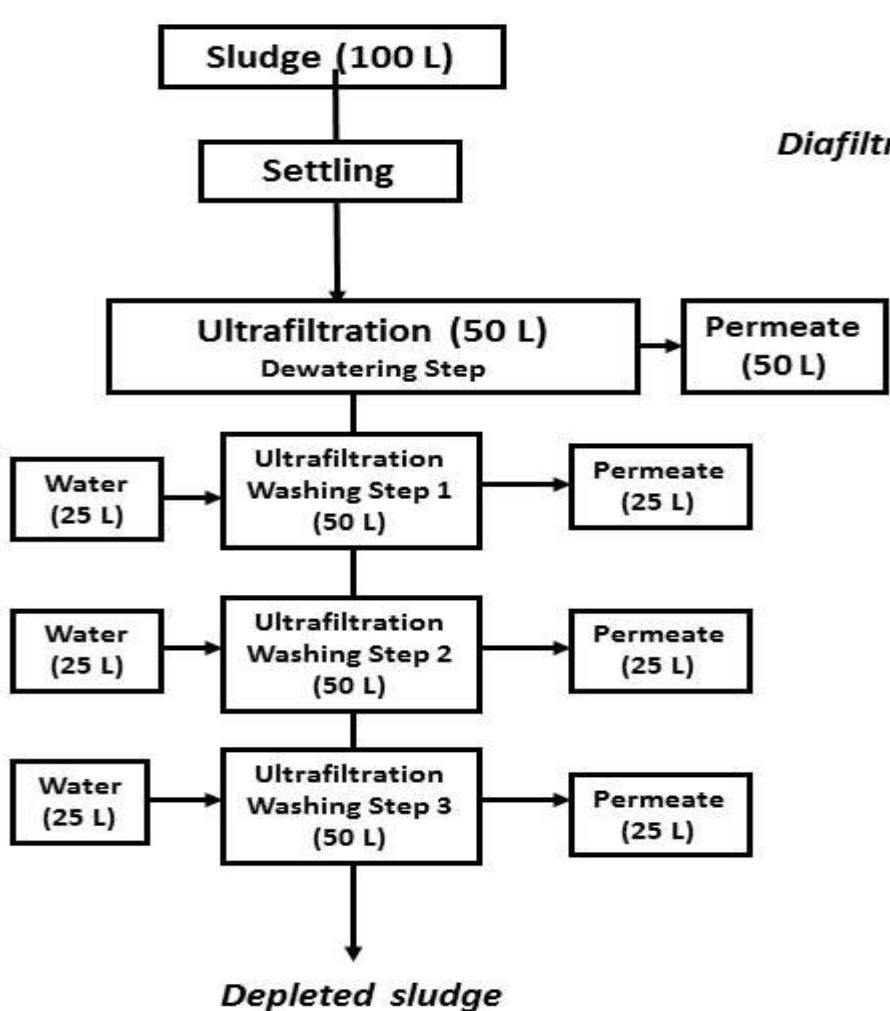
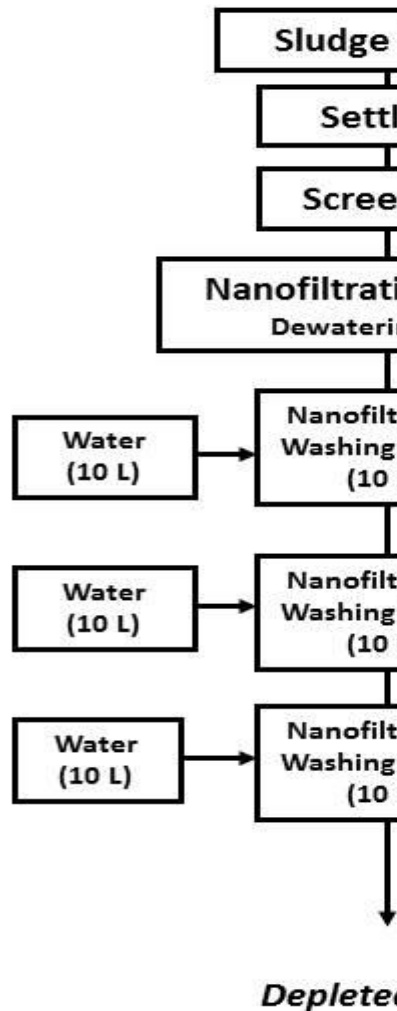


Fig. 2. Schematic diagram of pilot scale nanofiltration unit: [1] feed vessel (25 L), [2] temperature gauge [3] butterfly valve, [4] feed pump, [8] regenerative pump, [9] flow meter, [10] pressure gauge, [11] nanofiltration membrane, [12] pressure gauge [13] butterfly valve



A: Ultrafiltration Processing Scheme



B: Nanofiltration Processing Scheme

Fig. 3: Diafiltration Treatment Strategy for UF and NF processes (The measurements were made at a constant sludge volume fraction and a constant dilution step).

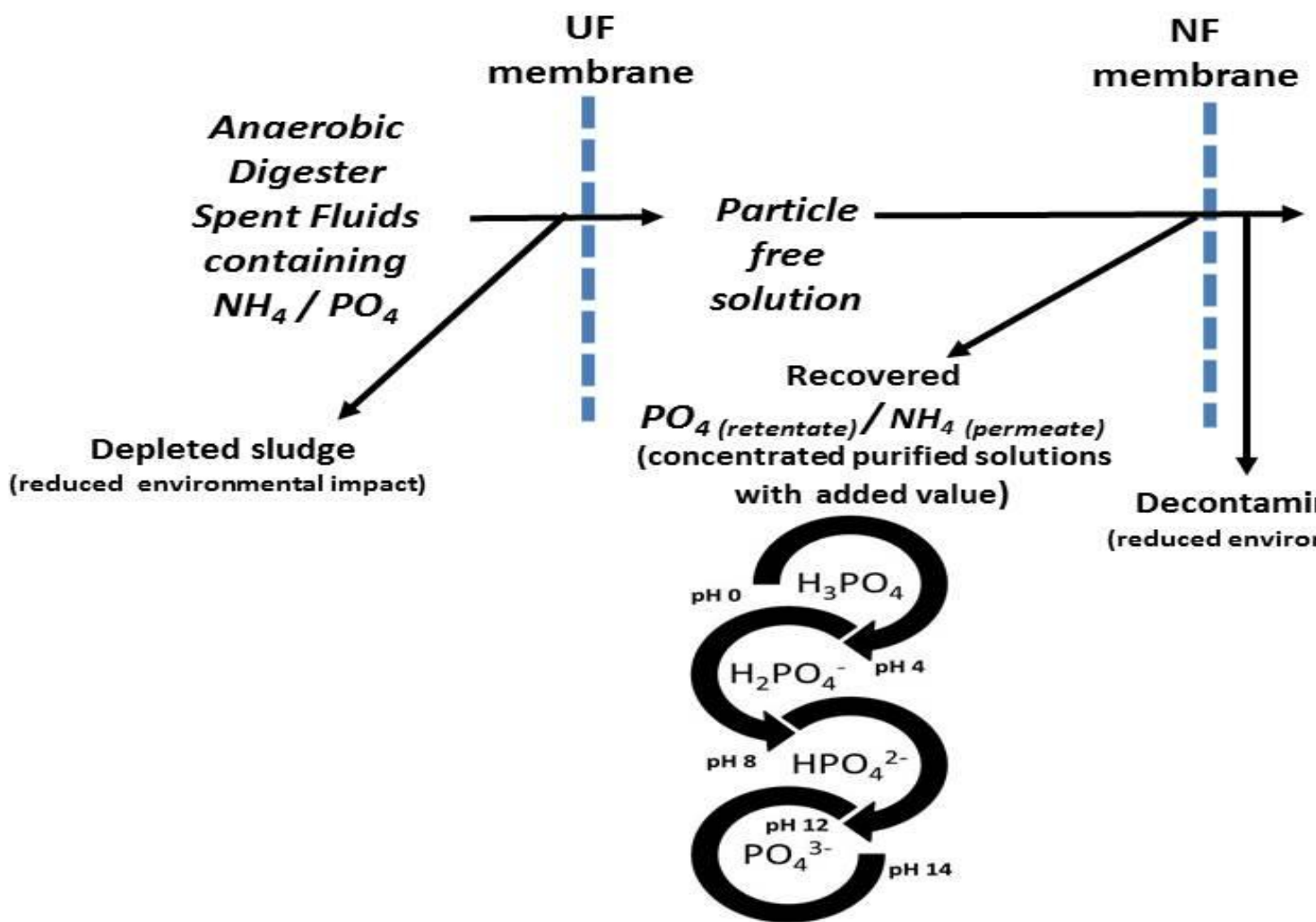


Fig.4. Separation scheme of phosphate and ammonia using UF and NF subsequently [28,29,31]

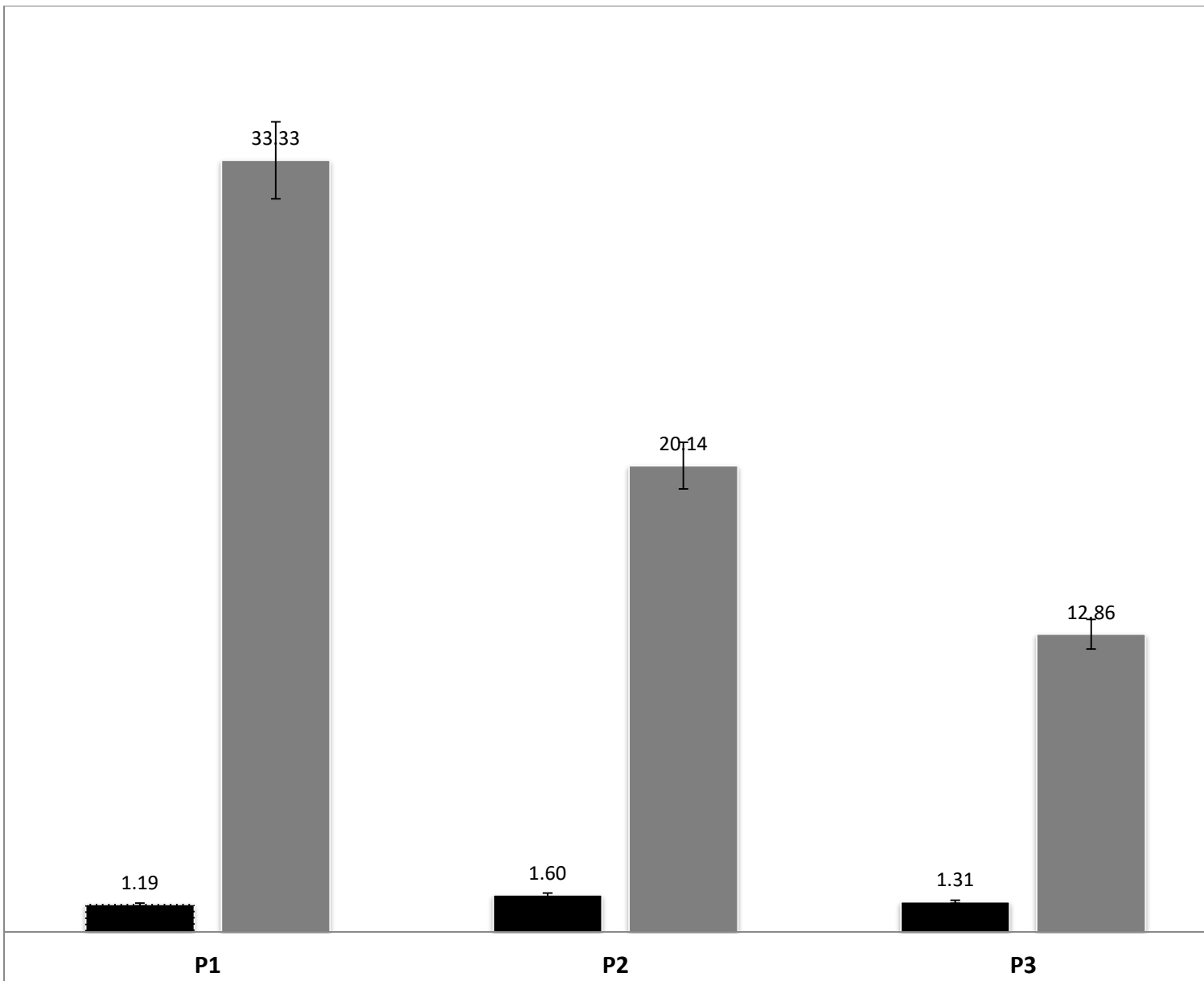


Fig. 5 : Concentration of phosphate (■) ammonia (■) (mmol L⁻¹) in the permeate during ultrafiltration (P1-P4 i.e. Permeate 1

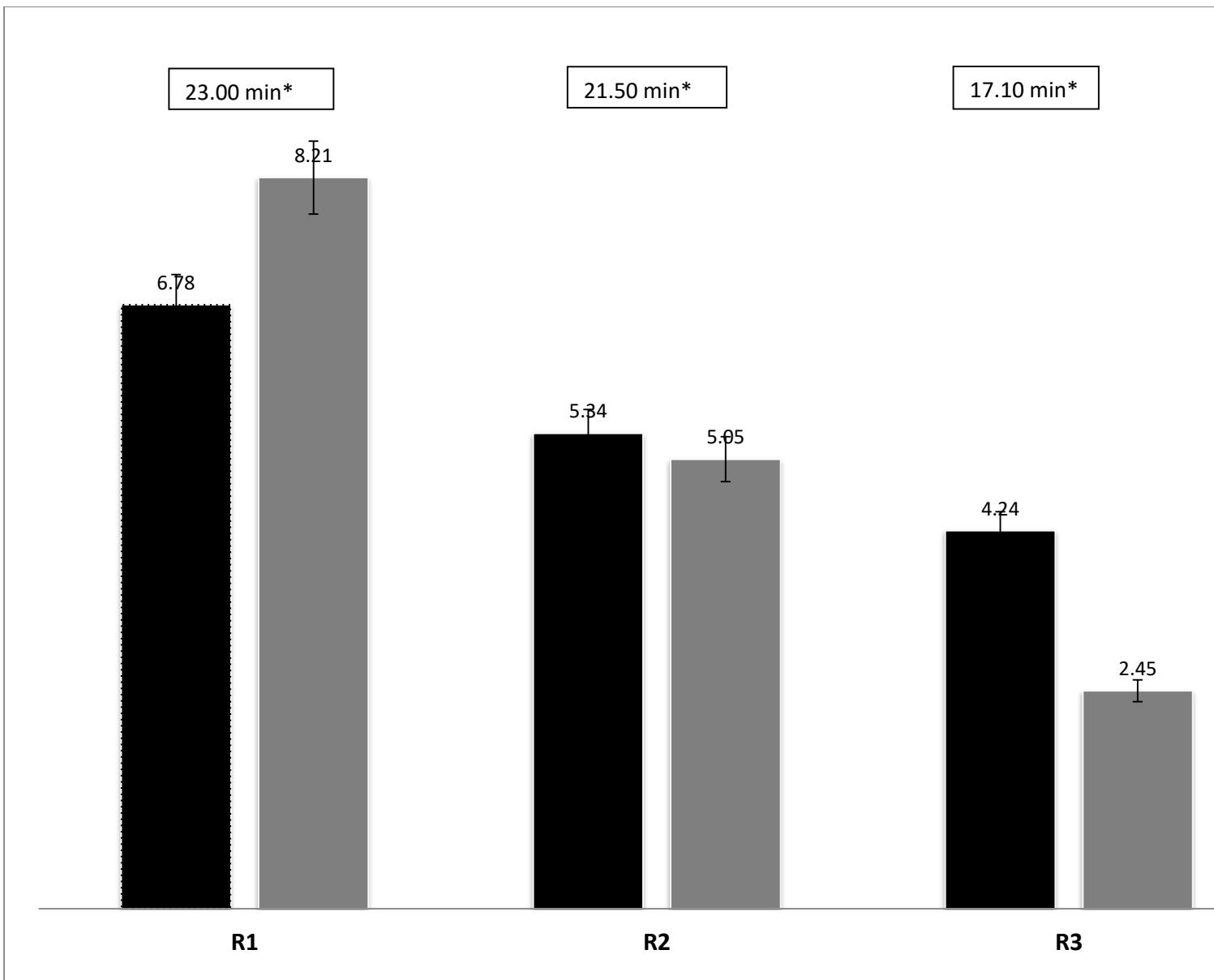


Fig .6 : Concentration of phosphate (■) ammonia (■) (mmols L⁻¹) in the retentate during ultrafiltration (R1-R4 i.e. Retentate 1-4)

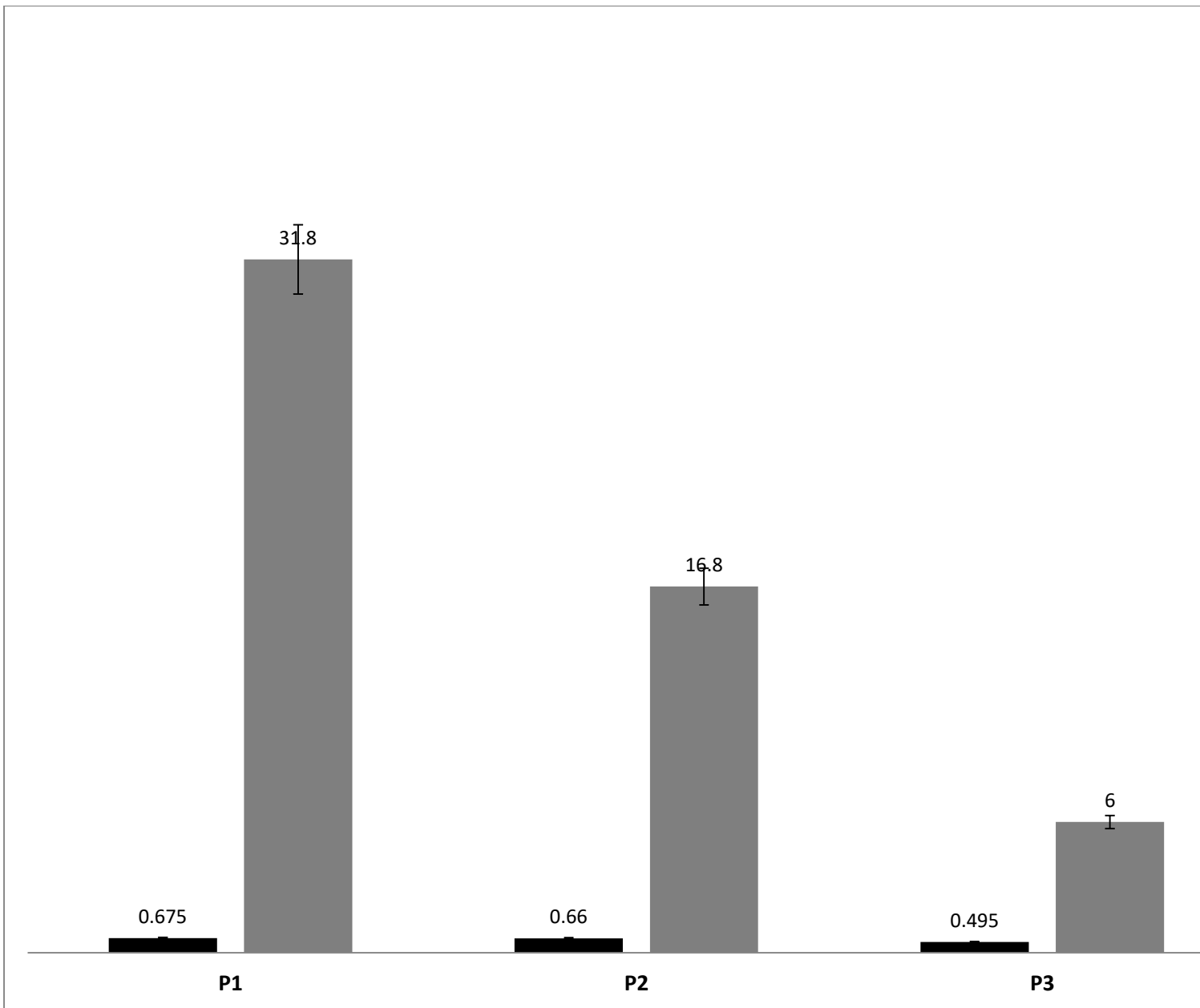


Fig.7: Concentration of phosphate(■) ammonia (■)(mmols L⁻¹) in the permeate during nanofiltration(P1-P4 i.e. Permeate 1-

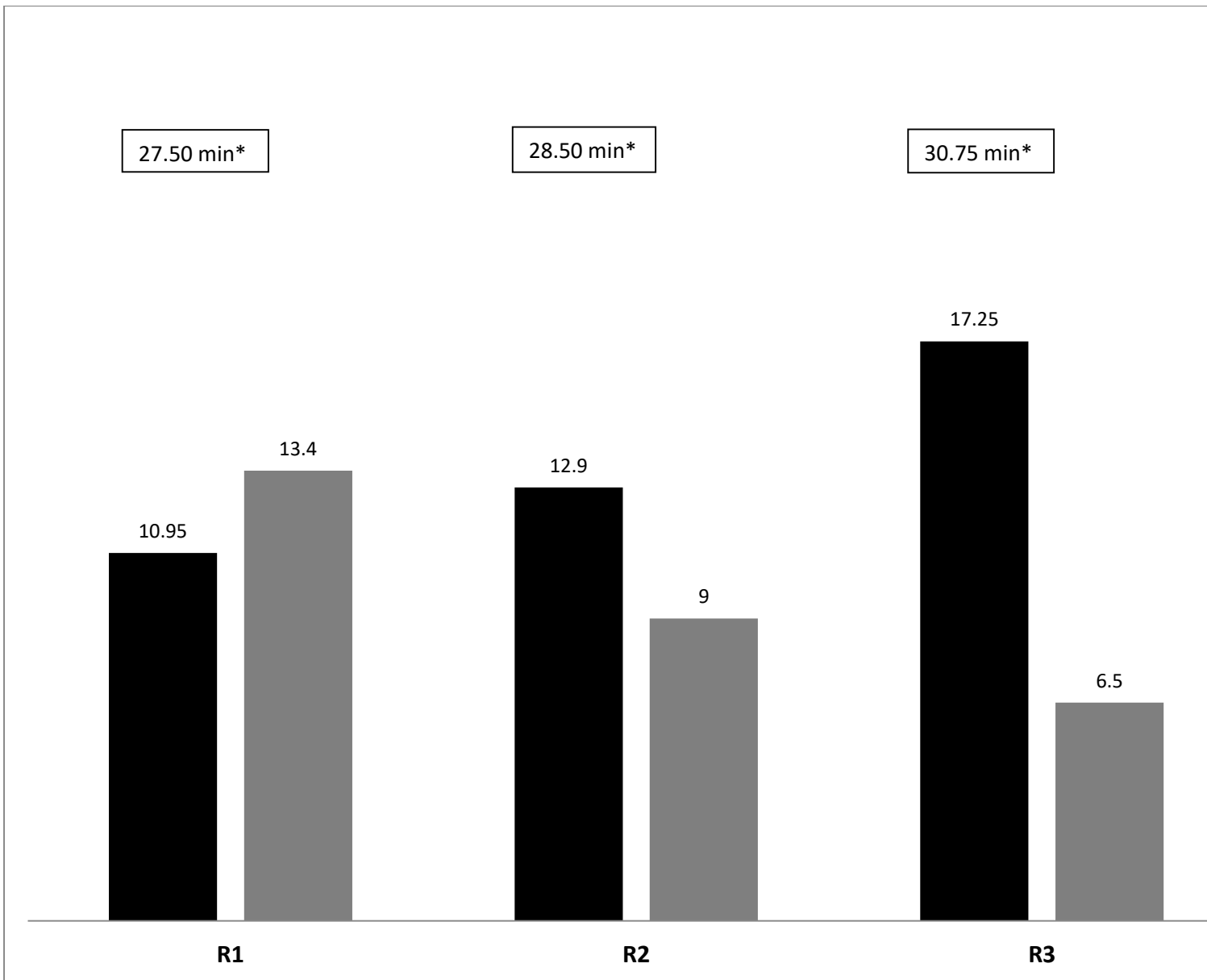


Fig.8.: Concentration of phosphate (■) ammonia (■)(mmols L⁻¹) in the retentate during nanofiltration(R1-R4 i.e. Retentate 1