



FEASIBILITY OF THE VALORIZATION OF A WASTE TITANIUM OXYCHLORIDE MIXTURE TO OBTAIN TITANIUM DIOXIDE

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INDEX

| | |
|---|----|
| INDEX | 3 |
| NOMENCLATURE..... | 4 |
| ABSTRACT..... | 5 |
| 1. INTRODUCTION | 6 |
| 1.1. WWTP's description..... | 7 |
| 1.2. R-1101 | 7 |
| 1.3. Sludge management at WWTP..... | 7 |
| 2. SCOPE AND SPECIFIC OBJECTIVES..... | 7 |
| 3. METHODS AND APPROACH | 8 |
| 3.1. Data analysis | 8 |
| 3.1.1. Incidences and reports. | 8 |
| 3.1.2. Sludge analytics. | 8 |
| 3.2. R-1101 reactions' study | 8 |
| 3.2.1. Precipitation reaction | 8 |
| 3.2.2. Neutralization reaction..... | 11 |
| 3.2.3. 1,3-Dichloropropanol's hydrolysis reaction. | 12 |
| 4. RESULTS AND DISCUSSION | 14 |
| 4.1. Preliminary mass balance | 14 |
| 4.2. Laboratory tests and analysis..... | 14 |
| 4.3. Industrial installation. | 14 |
| 4.3.1. Preliminary scale-up. | 14 |
| 4.3.2. Current installation's modifications..... | 14 |
| 4.4. Economic viability analysis..... | 14 |
| 5. CONCLUSIONS | 16 |
| 6. REFERENCES | 18 |
| 7. ANNEXES..... | 21 |
| 7.1. Plant's description..... | 21 |
| 7.2. Reactor R-1101 | 21 |
| 7.3. Daily reports 2023 | 21 |
| 7.4. Data analysis and preliminary conclusions..... | 21 |
| 7.5. Reactor R-1101 Study..... | 22 |
| 7.6. Laboratory session 1 | 22 |
| 7.7. Laboratory session 2 | 22 |
| 7.8. Laboratory session 3 | 22 |
| 7.9. Industrial Installation | 23 |
| 7.10. Economic viability analysis | 27 |

NOMENCLATURE

- AP:** Agricultural products, a BASF cluster.
ATEX: Explosive atmospheres.
CA: Cast iron (BASF nomenclature).
CAPEX: Capital expenditures.
CMR: Carcinogenic, Mutagenic, or toxic for Reproduction.
CSTR: Continuously stirred tank reactor.
DCP: 1,3-dichloropropan-2-ol.
DCS: Distributed control system
DOC: Dissolved organic carbon.
ED: Dispersions, a BASF cluster.
EPI: Epichlorohydrin.
HA: Stainless steel (BASF nomenclature).
LOI: Loss of ignition.
LE: Polypropylene (BASF nomenclature).
LD: Polyethylene (BASF nomenclature).
OPEX: Operational expenditures.
P&ID: Piping and instrumentation diagram.
PFD: Process flow diagram.
PDH: Propane dehydrogenation.
PLC: Programmable logic controller.
PP: Polypropylene
PVDF: Polyvinylidene fluoride
SEM: Scanning electron microscopy.
SHE: Security health and environment
TDS: Total dissolved solids.
WWTP-UT: Wastewater treatment plant and utilities.
XDR: X-ray diffraction.

ABSTRACT

The Waste-Water Treatment Plant (WWTP) at BASF Tarragona processes a --- kg·h⁻¹ stream characterized by high concentrations of **mixture A**. This stream undergoes neutralization in a Continuous Stirred Tank Reactor (CSTR), resulting in a biphasic solution comprising ----. This study initially focuses on the extraction, observation, and characterization of the reactor's product. Alongside, an examination of the mixture's potential value and the operational challenges faced by the plant.

Mixture A serves as a precursor in the production of----. This oxide is the most widely used white pigment globally, and a photocatalyst for water purification. Neutralization in the reactor yields ----, which, upon calcination between 400 and 800°C, transforms into ----. Approximately ---- kg/h of **product A**, generated during this process, is presently discarded alongside sludge. The high --- ions' content in this byproduct currently hinders its commercial viability. This highly --- mixture increases the TDSs (Total Dissolved Solids) of the sludge, requiring ----. Additionally, ---- compounds corrode the wetted parts of the plant's equipment.

Notably, €--- was expended on sludge stabilization in 2023 alone. In additions, several equipment processing the R-1101 mixture showed excessive corrosion problems.

This project investigated a potential ion cleaning processes, conducting lab-scale trials at the WWTP laboratory to tailor specific ionic effects. Concurrently, market research and engagement with stakeholders aided in identifying realistic constraints for commercialization. **Company A**, a --- consuming company, analyzed and initially accepted the delivered samples.

Based on the findings, a series of modifications to the plant's infrastructure and a new purification scheme for --- were proposed. This includes basic engineering for piping and equipment sizing, as well as strategies such as condensate recycling, reutilization of decommissioned equipment, and efficient space utilization.

An economic viability analysis estimated the project's capital and operational costs within a range of +/-30% certainty, capital cost ascended to ----€.

The analysis computed cash flows of ---- € considering only the savings from ---- (excluding potential revenue from **product A** sales). The capital investment recovery period would be of less than a year.

1. INTRODUCTION

The Wastewater Treatment Plant (WWTP) is located at BASF's Tarragona site. It processes the wastewater from nearly all plants within the site.

Approximately ---- tons of sludge are generated annually at the WWTP. In 2023 most sludge batches required ----, leading to a significant increase in operational costs. The **mixture A** might be contributing to the production of ----. Additionally, various recurring operational challenges may be related to the **mixture A**.

The byproduct of the **reactions** could potentially be a valuable product. However, it is currently treated as a waste stream, causing difficulties in the ----- treatment process.

1.1. WWTP's description.

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1.2. R-1101

-

1.3. Sludge management at WWTP

Sludge is managed in most large-scale wastewater treatment plants. Depending on the origin of the water, and the type of treatment, sludge may be disposed very differently. It may be fermented in bioreactors to obtain biofuel. It can also be incinerated, used as a fertilizer, or used for the concrete industry^[27]. Sludge might also not be suitable for revalorization and even require post treatments before its disposal into landfills.

In WWTP-UT three type of “sludges” can be differentiated.

These three sludges are currently mixed and thickened together up to 30-35% dry matter in centrifuges.

Table 1.3.2 Limit parameters for waste sludge landfilling in Catalonia.^[6]

| Parameter | Landfill limitation (Royal Decree 69/2009) |
|----------------------|--|
| TDS (mg/kg) | 60 000 |
| Chlorine (mg/kg) | 15 000 |
| DOC (mg/kg) | 800 |
| Loss at 105°C (%) | 65 |
| LOI (%) ¹ | 15 |

¹ Percentage over dry matter.

Most parameters can be exceeded due to exceptions established on the Royal Decree 69/2009. However, there is no flexibility regarding TDSs, over the limit, the whole pack must be stabilized by a third company.

2. SCOPE AND SPECIFIC OBJECTIVES

This project aims to provide BASF Española with a plausible solution/alternative to the sludge treatment problematics and its excessive commissioning costs. Focusing on the R-1101 effluent, its characteristics and its potential to be revalorized. The solution shall be simple and realistic, considering the plant's capacity and budget.

The specific objectives are:

- Confirm the cause-effect relationship between R-1101 effluent and certain plant's problematics. Determine de degree of affectation.
- Gather specific bibliographic information of the reactions that undergo to form the R-1101 sludge. Get acquainted with the affectation of different reacting conditions.
- Characterize the different sludges, analyze their composition and determine their potential value.

- Estimate the dimension of an installation suitable to perform a potential revalorization. Try to consider reusing part of the existing equipment and resources.
- Estimate the capital and operational costs of the proposed installation.
- Find potential stakeholders interested in financing the project and/or purchasing the revalorized product.

3. METHODS AND APPROACH

First, data analysis was performed to the relevant parameters to the whole **mixture A** treatment process, from inlet solution to the final sludge. The operational problems reported on 2023 were also screened to identify the ones potentially caused by the titanium oxychloride solution.

Second, an investigation of the sludge compounds and their properties, followed by bibliographical research on the reactions undergoing inside the R-1101. The plant's data on the reaction's performance was compared to theoretical and empirical data found. A hypothesis of the outlet's composition was presented.

The information obtained gave a more critical overview of the magnitude of the titanium oxychloride affectation as well as the magnitude of its potential value. It also provided a starting approximation of the mass balance of the reacting species to be expected in the R-1101. This approximation shed light to the preparation of experimental testing.

Finally, laboratory testing to discover the possible ways of purifying the raw sludge and its technical difficulties. Laboratory testing gave an overview of the resources needed to revalorize the R-1101 sludge.

3.1.Data analysis

In this section, historical data on the plant's corrective maintenance was revised. Repeated and (considered) relevant problematics were highlighted.

Historical data on residues' analytics and process variables were also revised.

3.1.1. Incidences and reports.

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3.1.2. Sludge analytics.

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3.2.R-1101 reactions' study

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3.2.1. Precipitation reaction

Titanium oxychloride is not a common, commercialized substance. It has no other source or application than being the result of TiCl_4 hydrolysis, and a precursor to possibly produce TiO_2 . The solution of titanium oxychloride (CAS 92344-13-3) is characterized in technical data sheets as an aqueous acidic solution with a 2:1 molar ratio of HCl to TiOCl_2 ($\text{TiOCl}_2 \cdot x \cdot \text{HCl}_y \cdot \text{H}_2\text{O}_z$). No information about pure TiOCl_2 solutions has been found.

TiOCl₂ is one possible intermediate in the TiCl₄ oxidation, depending on the reacting conditions. The most common and studied route for the TiCl₄ oxidation, and TiO₂ formation, is the chloride process (*Dupont, 1958*) consisting of gas phase high temperature (1200-1700°C) reactions where TiO₂ precipitates in an oxygen plasma or flame. This methodology and its kinetics were deeply studied by Wang et al., 2010^[13]. The most favored reaction route is displayed below:



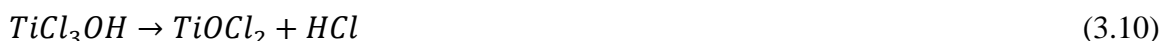
The formation of TiO₂ is the complete oxidation of TiCl₄. Kinetic data is predicted from TST (transition state theory) and RRKM (Rice–Ramsperger–Kassel–Marcus) theory^[13].

Table 3.2.3. Kinetic constants of the chloride process' reactions.

| Reaction | K _{298 K} (cm ³ /(mole·s)) | K _{700 K} (cm ³ /(mole·s)) | K _{1500 K} (cm ³ /(mole·s)) |
|----------|--|--|---|
| 3.4 | 2.0 × 10 ⁻³ | 3.0 × 10 ² | 5.0 × 10 ⁴ |
| 3.5 | 5.1 × 10 ⁻⁴ | 3.4 × 10 ¹ | 4.3 × 10 ³ |
| 3.6 | 4.8 × 10 ⁻³ | 8.4 × 10 ¹ | 6.1 × 10 ³ |
| 3.7 | 3.5 × 10 ⁻³ | 1.3 × 10 ² | 1.3 × 10 ⁴ |

At the conditions of the chlorine process, complete oxidation shall proceed easily due to the order of magnitude of the reaction rates.

TiOCl₂ formation pathway in a is also kinetically studied by Wang et al.



The reaction 3.10 seems to be highly unfavored in the forementioned process due to its endothermicity. instead, *TiCl₃OH* would more likely react with another water molecule as in reaction 3.5.

When TiCl₄ is hydrolyzed in an aqueous solution and at close-to-room temperatures (as in a sol-gel process¹) TiOCl₂ and HCl Lewis acid-base complex formation could be more favored.

¹ **Sol-gel:** Solid particles processing method. Monomers undergo polycondensation forming a colloidal solution with gel texture. The solution is then heated up to dehydrate the crystals formed^[4].

Shon et al. (2009b) ^[11] commented on the partial hydrolysis of TiCl_4 and presents TiOCl_2 as the main intermediate and precursor of hydrous titanium dioxide.

Ellis et al. (1976) ^[17] and *Einaga (1979b)* ^[18] deeply studied the dependence on pH of the formed titanium complexes, when TiCl_4 is partially hydrolyzed at room temperature. TiCl_4 is usually hydrolyzed in water or in an aqueous acid solution. Depending on the concentration of protons in the solution $[\text{H}^+]$ the complexes formed might tend to $[\text{TiO}]^{2+}$ or $[\text{Ti}(\text{OH})_2]^{2+}$.

The kinetic studies of *Ellis et al. (1976)* ^[17] (using a perchloric acid solution) state that above 0.1 mol dm^{-3} , $[\text{TiO}]^{2+}$ is vastly favored, therefore, TiClO_2 could be the obtained instead of $\text{TiCl}_2(\text{OH})_2$, obtained in the chloride process (equation 3.5). *Einaga (1979b)* ^[18] partially hydrolyzed TiCl_4 in a aqueous solution of $[\text{H}^+] = 0.5 - 4 \text{ mol dm}^{-3}$, it agreed on the favored formation of $[\text{TiO}]^{2+}$ complexes. *Gan et al. (2021b)* ^[5] also confirms the dominance of $[\text{TiO}]^{2+}$ in an acidic media:

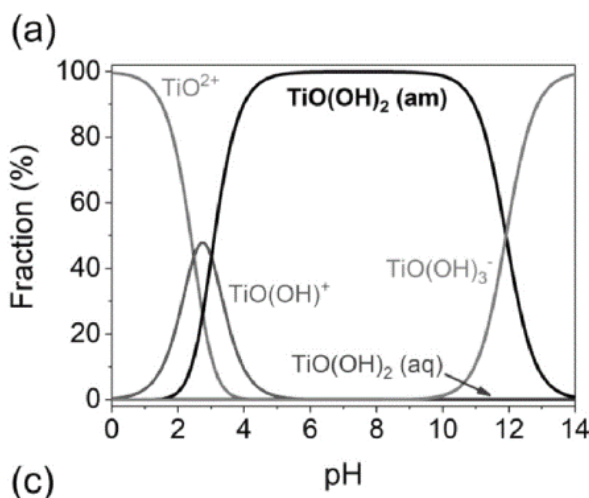


Figure 3.2.1. Titanium complex predominance over pH (*Gan et al., 2021*) ^[5].

This confirms the predominance presence and predominance of TiOCl_2 , however, when pH is increased, the study exclusively considers $\text{TiO}(\text{OH})_2$ structures (instead of $\text{Ti}(\text{OH})_4$) as the complete hydrolysis pathway for TiCl_4 . $\text{TiO}(\text{OH})_2$ monomers and polynuclear polycations such as $[(\text{TiO})_8(\text{OH})_{12}]$.

Other studies suggested different hydroxy monomeric complexes at higher pH ^{[2][14]}. $\text{Ti}(\text{OH})_4$ is commonly presented as the predominant species at pH higher than 6 (coinciding with the complex presented at equation 3.7).

Kotsyubynsky et al. (2016) ^[15] performed hydrolysis of TiCl_4 in a hydrochloric acid solution (36% in water) at room temperature. pH of one sample was increased to 8 with sodium hydrogen carbonate, while the other sample was maintained in the acidic media. The study suggests that reactions 3.4 to 3.7, are also expected in aqueous, room-temperature conditions.

The sample maintained in acidic media (pH close to 1) formed $[\text{Ti}(\text{OH})_2(\text{OH}_2)_4]^{2+}$ octahedral monomers. The sample with a pH raise up to 8 formed $[\text{Ti}(\text{OH})_4(\text{OH}_2)_2]^0$ monomers. The monomer consists of the favored structure $(\text{Ti}(\text{OH})_4)$ ^{[2][14]}, with the addition of two molecules of water to form the octahedral structure.

The forementioned studies partially agree in most content and yet have discrepancies on the exact reaction pathway and monomeric forms of the titanium precipitates. The transformation of TiOCl_2 into $\text{Ti}(\text{OH})_4$ (equation 3.1) might not be the most favored pathway. Regardless of the specific hydroxyl complexes formed, all studies agree on the formation of titanium dioxide when hydrolyzing TiCl_4 and furtherly calcinating it. The molar balance for the HCl and TiO_2

formation would not change regardless of the intermediate molecule. For this study, it is considered that $\text{Ti}(\text{OH})_4$ structures are favored at pH range of 6-11.

Titanium hydroxide is known to form an octahedral structure [24]. Ti^{4+} arranged with six oxygen atoms from OH^- and H_2O to form this structure. When calcinated residual water evaporates and the crystal structure remains with Titanium and oxygen. Monomers might arrange to form anatase, rutile, or brookite.

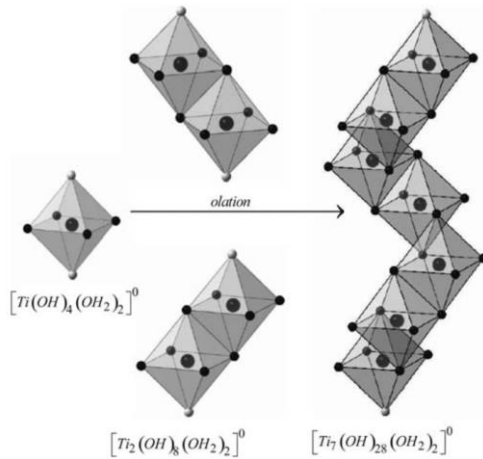


Figure 3.2.3. Titanium hydroxide olation (*Kotsyubynsky et al., 2016*) [15].

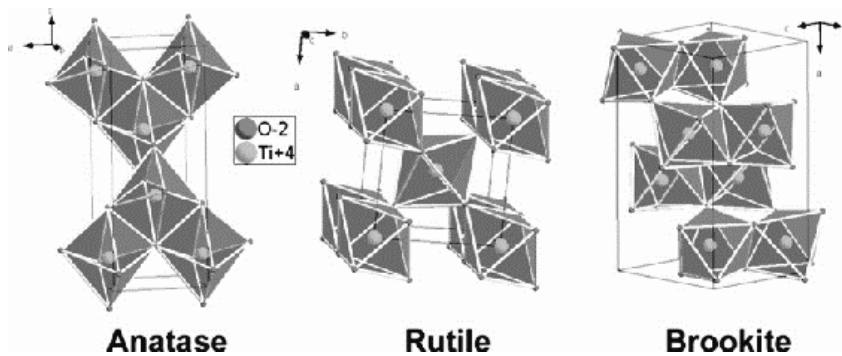


Figure 3.2.3. Ti complexes' possible distributions in octahedral structure (*Shalan et al., 2018*) [33].

In acidic media, $[\text{Ti}(\text{OH})_2(\text{OH}_2)_4]^{2+}$ monomers tend to arrange (when calcinated) in rutile structure, forming rod-like agglomerations. $[\text{Ti}(\text{OH})_4(\text{OH}_2)_2]^0$ may be arranged in anatase and/or brookite form, it shifts to a favored structure when calcinated at a specific temperature. Temperatures around 400-600 °C [28] shall favor the formation of anatase.

No kinetic models or computed rate constants were found for the aqueous process at moderate temperatures. In addition, several studies highlighted the lack of kinetic research in this matter. The experimental sessions of all studies reviewed report fast, semi-instantaneous precipitation of titanium complexes [18][11].

3.2.2. Neutralization reaction.

According to the definition of pH:

$$pH = -\log_{10}[\text{H}^+] \quad (3.11)$$

The proton concentration $[H^+]$ would have to decrease to the order of 10^{-7} by the neutralization with a base.

Hydrochloric acid is considered a strong acid due to its dissociation strength; its equilibrium constant (K_a) is approximately $1 \cdot 10^6$ ^[29]. For its neutralization, a strong base, sodium hydroxide is used. The dissociation constant (K_b) of sodium hydroxide is $6.3 \cdot 10^{-1}$.



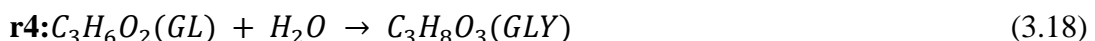
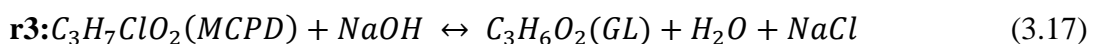
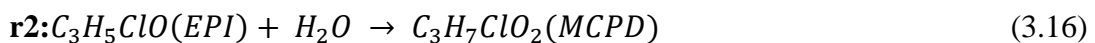
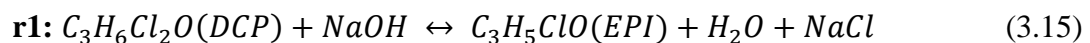
Perfect dissociation of the species (HCl and NaOH) in water was assumed. Self-ionization of water was neglected^[25]. According to (*Eigen & De Maeyer, 1955*) the kinetic constant for the neutralization (equation 3.14) is around $1,3 \cdot 10^{11}$ liter·mol⁻¹·sec⁻¹ (at 25°C).

3.2.3. 1,3-Dichloropropanol's hydrolysis reaction.

The DCP (1,3-dichloropropan-2-ol) is an halogenated, organic compound. It is an important intermediate in organic chemical industry. It can also be naturally produced in food by the reaction of fatty acids with chloride ions^[12]. This molecule is highly studied by its health impact, *Williams et al.*^[12], 2010 performed several tests on rats confirming DCP's carcinogenic category.

There are several studies involving DCP as an intermediate in the production of EPI (epichlorohydrin) from glycerin. The forementioned production process is a common procedure to reuse the spare glycerin obtained in the production of biodiesel^[10]. Kinetic studies on the reaction path of DCP are commonly fixated on the production of EPI using specific catalysts^[7] or have simplifications like the assumption of pseudo first order reactions^[8]. However, in 2022, a complete kinetic study on how DCP shifts to intermediates due to changes in pH^[9], was released. The study used HCl and NaOH as strong acid and base to shift Ph.

The path to hydrolyze DCP to GLY with NaOH is described by these four reactions in series:



DCP, EPI and MCPD are categorized as carcinogenic substances by the INSST (groups 1B, 1B and 2B respectively). GL is considered carcinogenic, mutagenic and reprotoxic. Glycerin is the only one considered non-CMR.

To attain the declassification of the CMR stream, incoming DCP might be oxidized to a non-CMR substance (glycerin).

Table 3.2.4. Kinetic constants of the DCP and derivate molecules' reactions. (Tollini *et al.*, 2022)

| Kinetic constant | A | Ea [J/mol K] |
|---------------------------|--|---------------------|
| $k_{r1(\text{alkali})}$ | 1,47E+11 [L/mol·s] | 6,60E+04 |
| $k_{-r1(\text{acid})}$ | 2,05E+09 [L/mol·s] | 6,70E+04 |
| $k_{-r1(\text{neutral})}$ | 4,95E+01 [L ² /mol ² ·s] | 4,80E+04 |
| $k_{r2(\text{acid})}$ | 2,02E+08 [L ² /mol ² ·s] | 7,50E+04 |
| $k_{r2(\text{neutral})}$ | 2,89E+01 [L ² /mol ² ·s] | 4,90E+04 |
| $k_{r2(\text{alkali})}$ | 5,24E+09 [L ² /mol ² ·s] | 8,80E+04 |
| $k_{r3(\text{alkali})}$ | 1,10E+19 [L/mol·s] | 1,20E+05 |
| $k_{-r3(\text{neutral})}$ | 2,68E+03 [L ² /mol ² ·s] | 7,00E+04 |
| $k_{-r3(\text{acid})}$ | 1,61E+08 [L/mol·s] | 5,70E+04 |
| $k_{r4(\text{acid})}$ | 3,42E+05 [L ² /mol ² ·s] | 6,30E+04 |
| $k_{r4(\text{neutral})}$ | 2,77E+03 [L ² /mol ² ·s] | 6,50E+04 |
| $k_{r4(\text{alkali})}$ | 3,42E+05 [L ² /mol ² ·s] | 6,30E+04 |

The kinetic parameters presented, and the tests conducted in the study ^[9] show that DCP is vastly predominant in a highly acidic environment. At moderate pH, EPI and MCPD might form, depending on the reagent concentration (reactions r1 and r2). At highly alkali pH (around 12) GL and GLY formation reactions are enhanced (reactions r3 and r4). Under a pH o 11 barely any GL or GLY should be formed. More specific calculus about DCP presence and reactivity inside the R-1101, are explained afterwards.

4. RESULTS AND DISCUSSION

Laboratory tests of the R1101 outlet were performed to characterize the stream. The potential of the stream was to be better understood, for both process disturbance and possible revalorization. The purification of the possible byproduct was studied in order to propose an industrial scale solution.

Several target companies for possible byproduct suppliace were contacted to analyze the markets demand and interest. Samples were sent to obtain specific feedback and a personalized offer.

A real scale installation was proposed for the purification of titania (TiO₂). Context of the available installation and services are considered in order to present the most economic and suitable option.

An economic viability analysis was performed considering the expected CAPEX and OPEX of the proposed installation.

Finally, based on all the data gathered, a recommendation of investment is presented, followed by out-of-scope next steps recommended for the project.

4.1.Preliminary mass balance

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4.2.Laboratory tests and analysis.

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4.3.Industrial installation.

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4.3.1. Preliminary scale-up.

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4.3.2. Current installation's modifications.

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4.4.Economic viability analysis.

Considering all data gathered, approximate cash flows are computed. Cash flows are calculated as follows:

$$\text{Cash Flow} = \text{net margin} + \text{depreciation} \quad (4.26)$$

$$\text{Net Margin} = \text{gross margin} - \text{fixed costs} - \text{depreciation} \quad (4.27)$$

$$\text{Gross margin} = \text{revenues} - \text{variable costs} \quad (4.28)$$

No fixed costs are considered in this analysis.

The capital cost is estimated by the sum of assembled equipment ([annex 7.10.2](#)), piping ([annex 7.10.1](#)), restoring ([annex 7.10.3.](#)), engineering and supervision costs ([annex 7.10.4](#)).

The variable costs are the utilities costs, displayed in [annex 7.10.5](#). The depreciation considered is a 10% of the total equipment cost, constant over the years up to 10 years ^[32]
Taking no benefits from the TiO₂ revalorization into account (TiO₂ transferred for free):

Table 4.4.6. Cash flows of a five-year period. No revenue from TiO₂ commercialization.

| Year | 0 | 1 | 2 | 3 | 4 | 5 |
|----------------------------|---------|--------|--------|--------|--------|--------|
| Capital Investment BASF | 321580 | | | | | |
| Revenues | | 721803 | 721803 | 721803 | 721803 | 721803 |
| Variable costs (utilities) | | 133502 | 133502 | 133502 | 133502 | 133502 |
| Gross margin | | 588301 | 588301 | 588301 | 588301 | 588301 |
| Depreciation | | 8738 | 8738 | 8738 | 8738 | 8738 |
| Margin before taxes | | 536303 | 517763 | 517763 | 517763 | 517763 |
| Net margin | | 402227 | 388322 | 388322 | 388322 | 388322 |
| Cash Flow | -321580 | 89385 | 397060 | 397060 | 397060 | 397060 |

Initially, a positive profit margin was expected only by saving the disposal costs of third parties. The IRR (Internal Rate of Return) is less than a year. From this starting point, if the byproduct can be sold for (hypothetically) 0.5 €/kg of dry TiO₂, the cash flows would raise like so.

Table 4.4.7. Cash flows of a five-year period. With revenue from TiO₂ commercialization.

| Year | 0 | 1 | 2 | 3 | 4 | 5 |
|----------------------------|---------|---------|---------|---------|---------|---------|
| Capital Investment BASF | 321580 | | | | | |
| Revenues | | 1413003 | 1413003 | 1413003 | 1413003 | 1413003 |
| Variable costs (utilities) | | 133502 | 133502 | 133502 | 133502 | 133502 |
| Gross margin | | 1279501 | 1279501 | 1279501 | 1279501 | 1279501 |
| Depreciation | | 8738 | 8738 | 8738 | 8738 | 8738 |
| Margin before taxes | | 1227503 | 1208963 | 1208963 | 1208963 | 1208963 |
| Net margin | | 920627 | 906722 | 906722 | 906722 | 906722 |
| Cash Flow | -321580 | 607785 | 915460 | 915460 | 915460 | 915460 |

This initial estimation categorizes the project as economically viable.

5. CONCLUSIONS

This project emerged from several challenges encountered in the sludge treatment process at the Wastewater Treatment Plant (WWTP) in BASF Tarragona. In 2023, more than ---- euros were spent exclusively on sludge disposal. ---- problems affected the equipment of the plant, especially the equipment processing the downstream of the reactor R-1101. This reactor neutralizes a **mixture A** with the addition of ---.

The slurry generated supposedly contained **product A** a valuable product with a 22-million-dollar global market. The purpose of the study was to demonstrate and quantify the affectation of R-1101 slurry to the plant. Additionally, to determine the possibility and value of segregating the slurry stream to an independent **product A** recovery treatment.

After three laboratory sessions it was determined that:

The **mixture A** contains approximately 33% of pure **active agent of mixture A** that almost fully converts into aqueous **product A** inside the R-1101. At a desired **mixture A** load of ---- $\text{kg}\cdot\text{h}^{-1}$ and a fivefold ---- proportion, an approximate ---- proportion of 0.7 would neutralize the mixture. The outlet stream would contain a 4.9% of **product A** (72% in dry matter). The main contaminants would be ---- and --- at 12% and 14% respectively (in dry matter). Production could raise up to ---- $\text{kg}\cdot\text{h}^{-1}$ (--- ton per month) of **product A**.

Company A, a **product A** consuming company, stated the possibility of absorbing up to --- ton/month of recovered **product A**. The company's main constraint in the product specification was the --- concentration, with a limit of 0.7% in mass.

At a laboratory scale, performing a two-step dilution and filtration process with 50°C deionized water, ---- concentration could be reduced to 0,4% obtaining up to 90% **product A**. Being ---- in the order of 7%, ---- in the order of 1% and other ---- under 1%.

Several samples were initially validated by **company A**, however they needed more significant samples to perform industrial testing.

To obtain the forementioned production and specification, approximately 2.3 $\text{ton}\cdot\text{h}^{-1}$ of extra industrial water would be needed. At least 7 $\text{ton}\cdot\text{h}^{-1}$ of 50°C deionized water would be required. It is proposed to integrate 5 $\text{ton}\cdot\text{h}^{-1}$ of reused condensate (already at 70°C) and 2 $\text{ton}\cdot\text{h}^{-1}$ of actual deionized water to reduce costs.

An installation proposal was presented. It mainly consisted of a set of two decanter centrifuges with two intermediate stirred tanks with condensate inlets. Part of the existing installation of WWTP was meant to be restored and reused in order to save capital costs.

The capital cost of the proposed installation would ascend to 322 k€ +/- 30%.

By considering only the savings from sludge disposal (excluding potential revenue from **product A** sales), the expected annual cash flow is projected at 400,000 euros. The internal rate of return would be less than one year, indicating strong economic viability and a promising investment opportunity. **Company A** has shown sustained interest in the project's realization.

Several considerations fall outside the scope of this project. In order to exhaust other purification methods, to assure the viability of the product and to have a more solid idea of the capital cost, a series of next steps are presented below:

Consider the viability of using an organic base (like ammonium hydroxide) instead of ---, to avoid --- formation. The organic base would volatilize in the calcination step. The cost of organic bases is usually higher, and the amount needed would be higher than of ---. The nitrogen would probably thermally oxidize to NO_x so gaseous effluents could need special treatments like selective reduction.

Consider the effect of molecule agglomeration and packing on the purification step. Contaminants tend to get trapped inside porous structures such as $\text{Ti}(\text{OH})_4$. The stirring intensity in R-1101 and the filtration/centrifugation intensity should be furtherly studied. Consider also the addition of templates (protective colloids) as polyalcohol, PVF, PVA. These measures could help maintaining a smaller particle size distribution and reduce the contaminant trapping.

Perform a more accurate engineering, establishing definitive piping and looking for specific offers from equipment providers. A +/- 10% economic viability analysis taking into account the product packing and transporting logistics.

Negotiate with GRACE the possibility to cover (at least partially) the capital investment of the project. In exchange, the revenue obtained could cover part of the caustic soda's expenses for the R-1101 (that is currently covered by GRACE).

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7. ANNEXES

7.1.Plant's description

Figure A.7.1.1 WWTP Process flow diagram.

7.2.Reactor R-1101

Figure A.7.2.1 Layout of WWTP with highlighted R-1101 zone.

Figure A.7.2.2. R-1101 supporting platform scheme.

Figure A.7.2.3. Reactor R-1101 top exploded diagram.

Figure A.7.2.4. Reactor R-1101 exploded scheme.

Figure A.7.2.5. R-1101 P&ID diagram.

Figure A.7.2.6. Control room's flow chart of the R-1101.

Figure A.7.2.7. P&ID BASF standard.

Figure A.7.2.7. P&ID BASF standard.

Figure A.7.2.8. First page of the SHE review of the R-1101, first version.

Table A.7.2.1. Transmitters involving the R-1101 and their functions.

Table A.7.2.2. Actuators involving the R-1101 and their functions.

7.3.Daily reports 2023

Figure A.7.3.2. Diagram D-340.

Figure A.7.3.1. Diagram D-1217.

7.4.Data analysis and preliminary conclusions

Table A.7.4.1. Chlorine concentration vs time in WWTP sludge during 2023 season.

Table A.7.4.2. Conductivity vs time in WWTP sludge during 2023 season.

Figure A.7.4.1. Conductivity of the physical-chemical treatment's effluent, reported in SAP.

7.5.Reactor R-1101 Study

Figure A.7.5.1 GRACE's TiOCl_2 solution technical data sheet.

Figure A.7.5.2. GRACE's TiOCl_2 solution safety data sheet.

Figure A.7.5.3 BASF's TiOCl_2 solution analysis of organic compounds.

Table A.7.5.1. GRACE tests on TiOCl_2 solution's composition.

Table A.7.5.2. GRACE tests on TiOCl_2 solution's physical properties.

Figure A.7.5.4. R-1101 content's analysis for DCP concentration.

Figure A.7.5.5. Physical-chemical treatment outlet's analysis for DCP concentration.

Figure A.7.5.6. R-10D content's analysis for DCP concentration.

7.6.Laboratory session 1

Figure A.7.6.1. Irregular R-1101 conditions, oscillation of pH due to soda excess.

Figure A.7.6.2. *Agrolab*'s XDR analysis report for laboratory session 1 (page 1).

Figure A.7.6.3. *Agrolab*'s XDR analysis report for laboratory session 1 (page 2).

Figure A.7.6.4. *Agrolab*'s XDR analysis report for laboratory session 1 (page 3).

Figure A.7.6.5. *Agrolab*'s XDR analysis report for laboratory session 1 (page 4).

Figure A.7.6.6. *Agrolab*'s XDR analysis report for laboratory session 1 (page 5).

7.7.Laboratory session 2

7.8.Laboratory session 3

Figure A.7.8.1. R-1101 conditions, before sample extraction, for the third laboratory session.

Figure A.7.8.2 R-1101 conditions, before sample extraction, for the third laboratory session. Red for pH, green for industrial water, blue for soda and black for TiOCl_2 solution

Table A.7.8.1. Third laboratory session experiment parameters.

Figure A.7.8.3 XRD analysis of samples B, 5,6,7 and 9 (displayed in order). Analysis provided by *Torreid S.A.*

Figure A.7.8.4. TGA analysis of samples B, 5,6,7 and 9 (displayed in order). Analysis provided by *Torreacid S.A.*

Table A.7.8.2 Lost mass percentage at 200 and 900°C, according to TGA.

7.9.Industrial Installation

Figure A.7.6.1. Diagram SK-600282.

Figure A.7.6.2. Diagram SK-313.

Figure A.7.6.3. Diagram SK-1345.

Figure A.7.6.4. Diagram SK-793.

Figure A.7.6.5. Diagram SK-352.

Figure A.7.6.6. Diagram SK-1444.

Figure A.7.6.7. New condensate piping layout.

Figure A.7.6.8. New condensate piping layout.

Figure A.7.6.9. New installation piping layout. Blue: industrial air, orange: residual water from centrifuges, yellow: slurry from R-9A to centrifuges.

Figure A.7.6.10. New installation piping layout, new pump proposed. Blue: industrial air and yellow: slurry from R-9A to centrifuges.

Figure A.7.6.11. Proposed location for centrifuge system installation. Building A-12.

Figure A.7.6.12. Proposed condensate pipe layout (red line). Extracted from diagram N-

Figure A.7.6.13. Justification mail with Tarragona Power regarding condensate characteristics.

Figure A.7.6.14. Justification mail with Tarragona Power regarding condensate characteristics.

Figure A.7.6.15. Justification mail with Tarragona Power regarding condensate characteristics.

Figure A.7.6.16. Justification mail with Tarragona Power regarding condensate characteristics.

Figure A.7.6.17. SAP extract of pump *Wilden XPS830* availability at Tarragona's site warehouse.

SPEC SHEET **PROFLO**
PROGRESSIVE PUMP TECHNOLOGY

P400 PLASTIC PUMP

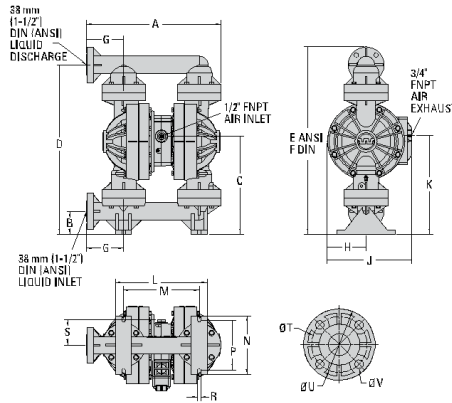
38 mm (1-1/2") Pump
Maximum Flow Rate:
454 lpm (120 gpm)



P400 PLASTIC

DIMENSIONAL DRAWINGS

P400 Polypropylene



DIMENSIONS

| ITEM | METRIC (mm) | STANDARD (inch) |
|------|-------------|-----------------|
| A | 476 | 18.8 |
| B | 81 | 3.2 |
| C | 348 | 13.7 |
| D | 602 | 23.7 |
| E | 665 | 26.2 |
| F | 677 | 26.6 |
| G | 131 | 5.2 |
| H | 138 | 5.4 |
| J | 300 | 11.8 |
| K | 351 | 13.8 |
| L | 324 | 12.8 |
| M | 268 | 10.6 |
| N | 208 | 8.2 |
| P | 176 | 6.9 |
| R | 12 | 0.5 |
| S | 91 | 3.6 |

DIN FLANGE

| | | |
|---|----------|----------|
| T | 110 DIA. | 4.3 DIA. |
| U | 150 DIA. | 5.9 DIA. |
| V | 18 DIA. | 0.7 DIA. |

ANSI FLANGE

| | | |
|---|----------|----------|
| T | 98 DIA. | 3.9 DIA. |
| U | 127 DIA. | 5.0 DIA. |
| V | 16 DIA. | 0.6 DIA. |

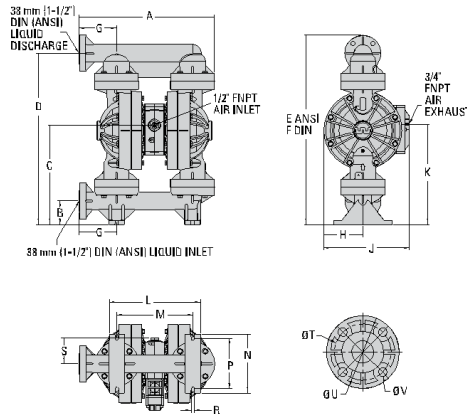
LW0324 REV. A

PUMP DATA

Specifications:

- Air Inlet..... Polypropylene 19 kg (41 lb)
- PVDF 27 kg (59 lb)
- Air Inlet..... 13 mm (1/2")
- Liquid Inlet..... 38 mm (1-1/2")
- Liquid Outlet..... 38 mm (1-1/2")

P400 PVDF



DIMENSIONS

| ITEM | METRIC (mm) | STANDARD (inch) |
|------|-------------|-----------------|
| A | 471 | 18.5 |
| B | 83 | 3.2 |
| C | 345 | 13.6 |
| D | 596 | 23.4 |
| E | 659 | 25.9 |
| F | 670 | 26.4 |
| G | 130 | 5.1 |
| H | 137 | 5.4 |
| J | 300 | 11.8 |
| K | 348 | 13.7 |
| L | 319 | 12.6 |
| M | 264 | 10.4 |
| N | 205 | 8.1 |
| P | 174 | 6.9 |
| R | 12 | 0.5 |
| S | 91 | 3.6 |

DIN FLANGE

| | | |
|---|----------|----------|
| T | 110 DIA. | 4.3 DIA. |
| U | 149 DIA. | 5.9 DIA. |
| V | 18 DIA. | 0.7 DIA. |

ANSI FLANGE

| | | |
|---|----------|----------|
| T | 96 DIA. | 3.9 DIA. |
| U | 126 DIA. | 5.0 DIA. |
| V | 16 DIA. | 0.6 DIA. |

LW0325 REV. A

WILDEN

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www.wildenpump.com



WIL-11241-T-01

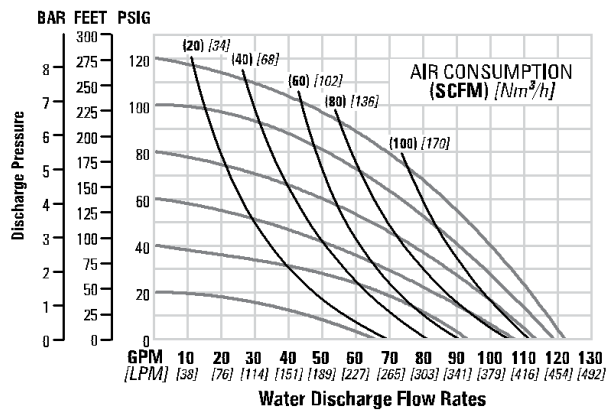
Figure A.7.6.18. Wilden P400 technical data sheet.

SPEC SHEET **PROFLO**
PROGRESSIVE PUMP TECHNOLOGY
P400 PLASTIC PUMP

PERFORMANCE

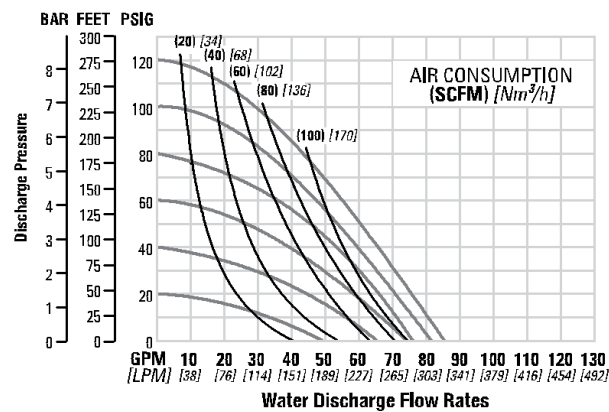
**P400 PLASTIC
RUBBER-FITTED**

Max. Flow Rate..... 454 lpm (120 gpm)
Max. Inlet Pressure..... 8.6 bar (125 psig)
Max. Solids Passage..... 6.4 mm (1/4")
Max. Suction Lift..... 5.5 m Dry (18.2')
9.0 m Wet (29.5')



**P400 PLASTIC
REDUCED-STROKE
PTFE-FITTED**

Max. Flow Rate..... 318 lpm (84 gpm)
Max. Inlet Pressure..... 8.6 bar (125 psig)
Max. Solids Passage..... 6.4 mm (1/4")
Max. Suction Lift..... 3.3 m Dry (10.8')
9.7 m Wet (31.8')



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a DUNIPER company
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Figure A.7.6.19. Wilden P400 technical data sheet, performance graphs.

7.10. Economic viability analysis

The justifications for the cost's estimation were agreed and discussed with the WWTP-UT engineering and maintenance manager on the 12th and 20th of February of 2024.

Piping estimation

Based on previous experiences in similar projects, the following cost estimation has been determined:

Table A.7.10.1. Piping costs' justifications.

Equipment estimation.

Based on previous experiences in similar projects. The different needs for each unit's installation are considered (E&I, structure, paving and so on). Control loops and their actuation valves are considered separately.

The following cost estimation has been determined:

Table A.7.10.1. Equipment costs' justifications.

Restoring of existing installation.

There are several installations that intended to be reused for the project's purposes.

The estimation of the restoration costs is displayed below:

Table A.7.10.1. Restoring costs' justifications.

Engineering and supervision costs.

The estimated duration for the civil work is set to 2 months, the supervision and engineering costs are displayed below (all duration estimations have been proposed by the project manager):

Table A.7.10.1. Engineering costs' justifications.

Variable costs (Utilities).

Deionized water, industrial water and condensate requirements are extracted from the preliminary scale-up, section 4.3.1. Industrial air consumption is estimated by the expected supply requirement for three pneumatic pumps of the selected models ([figure A.7.6.19](#)). Electricity is estimated by the approximate consumption of the existing centrifuges.

Table A.7.10.1 Variable costs' justification.



Oferta: 333806

Soluciones de Bombeo | tecnicafluidos.es

| Pos. | Artículo | Cantidad | Plazo | Precio ud | % Dto | Precio ud con Dto | Total |
|--|---|----------|------------------|-----------|-------|-------------------|---------------------|
| <i>Oferta de bombas Wilden P400 PP y P400 PVDF</i> | | | | | | | |
| 1 | 04-12745 P400/PPPPP/TWS/TF/PTV/0504 (C) BOMBA NEUMÁTICA WILDEN DE DOBLE DIAFRAGMA (Entregada con silenciador de escape) - Tamaño: 1 1/2" (brida DIN DN40 PN10/16) - Material partes en contacto con el líquido Cuerpo: POLIPROPILENO Membranas , bolas y juntas en TEFLON PTFE - Material válvula de aire: Polipropileno - Peso bomba: 19 Kg Incluído manual de O&M y CE | 1 | Inmediato s/vta. | 6.423,66 | 55,0 | 2.890,65 | 2.890,65 EUR |
| 2 | 04-13495 P400/KKPPP/TWS/TF/KTV/0504 (C) BOMBA NEUMÁTICA WILDEN DE DOBLE DIAFRAGMA (Entregada con silenciador de escape) - Tamaño: 1 1/2" (brida DIN DN40 PN10/16) - Material partes en contacto con el líquido Cuerpo: PVDF Elastómeros: Teflón Full Flow - Material válvula de aire: Polipropileno - Peso bomba: 27 Kg Incluído manual de O&M y CE | 1 | Inmediato s/vta. | 13.090,28 | 55,0 | 5.890,63 | 5.890,63 EUR |
| Total sin impuestos | | | | | | | 8.781,28 EUR |

Figure A.7.10.1 Wilden P400 offer from *Técnica de Fluidos*.

Figure A.7.10.2 Centrifuges S-6A/B consumption ^[30].