



Treball Final de Grau

Basic design of a polybenzimidazole (PBI) production plant

Laura Recacha Benito

June 2024



UNIVERSITAT DE
BARCELONA

Aquesta obra està subjecta a la llicència de:
Reconeixement–NoComercial–SenseObraDerivada



<http://creativecommons.org/licenses/by-nc-nd/3.0/es/>

The true delight is in the finding out rather than in the knowing.

Isaac Asimov

En primer lugar, a mis padres, por construir una realidad donde mi progreso académico solo se ha podido dañar por mis acciones, y nunca por mi entorno. A mi hermana y hermano, por el apoyo y los consejos. A mi perro y a todos ellos por la tranquilidad que me han dado cuando la he necesitado.

A mi amigo Esteban, sin el cual este trabajo no sería el mismo. Ha sido el empujón que me faltaba. Gracias a él, puedo recordar cada día de estos últimos meses con el cariño que les ha aportado.

A mis tutores, la Dra. Esther Chamarro Aguilera y el Dr. Manuel Vicente Buil, a quienes debo gran parte de mi aprendizaje en esta etapa. Agradezco su pericia, paciencia y dedicación.

Por último, a todas aquellas personas que han pasado por mi vida y le han dado forma durante estos años, por cada cambio que he vivido.

CONTENTS

SUMMARY	I
RESUM	III
SUSTAINABLE DEVELOPMENT GOALS	V
1. INTRODUCTION	1
1.1. Polybenzimidazole	2
1.2. Market study	7
2. OBJECTIVES	11
3. PATENT RESEARCH	13
4. PRODUCTION PROCESS	15
4.1. Raw materials	17
4.2. Block diagram	18
4.3. Mass balance	21
4.4. Recipe	25
4.4.1. Raw materials reception	25
4.4.2. Polycondensation reaction	26
4.4.3. Filtering	27
4.4.4. Drying	28
4.4.5. Milling & packaging	28
5. EQUIPMENT SPECIFICATION	31
5.1. Reactor vessel (V-01)	31
5.2. Humid mill (M-01)	33
5.3. Reactor vessel (V-02)	33
5.4. Bag filter (F-01)	34
5.5. Evaporator (V-03)	35
5.6. Dryer (DR-01)	35
5.7. Hopper (H-01)	36
5.8. Ball mill (M-02)	37

5.9.	Sieve and Packaging (SP-01)	38
5.10.	Plant services	39
5.11.	Additional equipment	39
6.	P&ID DIAGRAM	41
7.	PRODUCTION PLANNING	45
7.1.	Time study	45
7.2.	Scheduling	49
7.3.	Production campaigns	53
7.4.	KPIs determination	55
8.	CONCLUSIONS	57
	REFERENCES AND NOTES	59
	ACRONYMS	61
	APPENDICES	63
	APPENDIX 1: TABLE CONTENT	63
	APPENDIX 2: TABLE CONTENT	77
	APPENDIX 3: FIGURE CONTENT	79

SUMMARY

Polybenzimidazoles (PBIs) are a large family of engineering plastics comprised in a bigger category of polymers with benzimidazole groups as part of the structural repeat unit. PBIs can be partly or fully aromatic, and are typically characterized by outstanding thermo-mechanical properties. This makes them particularly suitable for various demanding high-temperature applications in the form of fibers, coatings, or membranes.[3]

The objective of this study is to perform the basic design of a polybenzimidazole (PBI) production plant, operating in discontinuous. A market study is performed to determine global production of the polymer, and the designed plant is set to represent a 1 % of that production. Therefore, an annual production of 1000 tonnes/year has been selected. The produced PBI has an average chain molecular weight of 30 000 Mw, a purity of 99 % and a particle size between 100 and 200 μm , and will be packed in big bags of 500 kg.

A recipe has been designed, based on existing processes found by a patent research. This recipe is detailed in writing and illustrated in the form of a block diagram.

Batch size is 5000 kg, for which a mass balance is carried out, to show the necessary raw materials consist of 3800 kg of 3,3',4,4'-tetraminobiphenyl (TAB) and 5650 kg of diphenyl isophthalate (DPIP).

The appropriate equipment has been carefully selected for each unit of the process. This consists of two reaction vessels where polymerization occurs (V-01, V-02), with a humid mill between them (M-01), followed by a bag filter (F-01), a vessel where evaporation of solvent and precipitation of product occur (V-03), and a dryer to remove all remaining humidity (DR-01). In addition, there is a ball mill (M-02) that corrects particle size, and, finally, a sieving and packaging unit (SP-01).

With the forementioned information, a P&ID is created, to display the process in depth.

A time study is conducted for every existing operation, determining thus the occupation time of each piece of equipment. The resulting batch time (BT) is 63 h, while cycle time (CT), conditioned by the bottleneck (vessel V-02), is 25 h.

Considering work conditions of the plant; 3 shifts, 24 h/day, 7 day a week, amounting to 6900 h a year, it has been proved that the proposed yearly production of 1000 tonnes is achievable with a total of 200 batches, configured in 20 campaigns of 10 batches.

The following KPIs have been calculated; a maximum number of batches of 274, the maximum production capacity of 1370 tonnes/year, and a minimum production time of 5038 h/year. The production planning of the plant allows for an additional 7 weeks where the line is available for a different process.

Necessary plant services include nitrogen for inertization, deionized water for cleaning, and thermal oil, heated in a boiler and cooled in a heat exchanger.

Keywords: Polybenzimidazole, PBI, polymer, basic design, production plant

RESUM

Els polibenzimidazols (PBI) són una gran família de plàstics d'enginyeria inclosos en una categoria més àmplia de polímers amb grups de benzimidazol com a part de la unitat repetitiva estructural. Els PBI poden ser parcialment o completament aromàtics, i es caracteritzen típicament per les seves excepcionals propietats termo-mecàniques. Això els fa especialment adequats per a diverses aplicacions exigents a alta temperatura en forma de fibres, recobriments o membranes [3].

L'objectiu d'aquest estudi és realitzar el disseny bàsic d'una planta de producció de polibenzimidazol (PBI), operant de manera discontinua. S'ha fet un estudi de mercat per determinar la producció global del polímer, i la planta dissenyada representa un 1 % d'aquesta producció. Per tant, s'ha seleccionat una producció anual de 1000 tones/any. El PBI produït té un pes molecular mitjà de cadena de 30.000 Mw, una puresa del 99 % i una mida de partícula entre 100 i 200 µm, i serà envasat en big bags de 500 kg.

S'ha dissenyat una recepta, basada en processos existents trobats mitjançant una recerca de patents. Aquesta recepta està detallada per escrit i il·lustrada en forma de diagrama de blocs. La mida del lot és de 5000 kg, per a la qual es realitza un balanç de masses, mostrant que les matèries primeres necessàries consisteixen en 3800 kg de 3,3',4,4'-tetraminobifenil (TAB) i 5650 kg de isoftalat de difenil (DPIP).

S'ha seleccionat l'equip adequat per a cada unitat del procés. Consisteix en dos reactors on es dona la polimerització (V-01, V-02), amb un molí humit entre ells (M-01), seguit d'un filtre de bosses (F-01), un recipient on ocorre l'evaporació del solvent i la precipitació del producte (V-03), i un assecador per eliminar tota la humitat restant (DR-01). A més, hi ha un molí de boles (M-02) que corregeix la mida de les partícules, i, finalment, una unitat de tamissat i envasat (SP-01).

Amb la informació esmentada, es crea un P&ID per mostrar el procés en profunditat.

Es realitza un estudi de temps per a cada operació existent, determinant així el temps d'ocupació de cada equip. El temps resultant per lot (BT) és de 63 h, mentre que el temps de cicle (CT), condicionat per l'equip limitant (V-02), és de 25 h.

Considerant les condicions de treball de la planta; 3 torns, 24 h/dia, 7 dies a la setmana, que sumen 6900 h a l'any, s'ha demostrat que la producció anual proposada de 1000 tones és assolible amb un total de 200 lots, configurats en 20 campanyes de 10 lots.

S'han calculat els següents KPIs; un nombre màxim de lots de 274, la capacitat màxima de producció de 1370 tones/any, i un temps mínim de producció de 5038 h/any. La planificació de producció de la planta permet setmanes addicionals on la línia està disponible per a un procés diferent.

Els serveis necessaris per a la planta inclouen nitrogen per a la inertització, aigua desionitzada per a la neteja, i oli tèrmic, escalfat en una caldera i refredat en un intercanviador de calor.

Paraules clau: Polibenzimidazol, PBI, polímer, disseny bàsic, planta de producció

SUSTAINABLE DEVELOPMENT GOALS

Decent Work and Economic Growth: The development of the project will introduce a market segment in the Spanish territory that currently has little presence, generating wealth and creating numerous jobs.

Industry, Innovation, and Infrastructure: The production plant will boost the chemical industry as well as research in the R&D department.

Gender Equality: The plant will have a zero-discrimination policy, promoting hires regardless of gender, thus helping to break the glass ceiling.

1. INTRODUCTION

For over 100 years, plastics have been present in the chemical industry, with their importance increasing steadily over time, due to their adaptability, lightweight, durability, and low production cost. Polymers, which make up all modern plastics, are large molecules consisting of repeating monomers.

In the early decades of the 20th century, alliances were formed by petroleum and chemical industries, giving a use to waste material from processing crude oil and natural gas. To this day, they are still the main producers of raw material resins for the plastics industry. [1]

Although plastic pollution poses a great environmental challenge, the importance of this industry can not be denied, with a yearly global production of more than 400 million metric tonnes. [2]

Plastics can be classified as commodity, engineering and high-performance plastics. The latter are those which offer superior mechanical, thermal, chemical and electrical properties that make them suitable for specialized applications. One of these polymers of significant interest is polybenzimidazole (PBI).

Polybenzimidazoles (PBIs) are a large family of engineering plastics comprised in a bigger category of polymers with benzimidazole groups as part of the structural repeat unit. PBIs can be partly or fully aromatic, and are typically characterized by outstanding thermo-mechanical properties. This makes them particularly suitable for various demanding high-temperature applications in the form of fibers, coatings, or membranes.[3]

The polybenzimidazole on which most attention has been focused over the past 25 years and, therefore, the one that is discussed hereinafter is poly[2,2'-(m-phenylene)-5,5'-bibenzimidazole]. It is the only commercially available polybenzimidazole and it is the specific structure that is typically intended when the acronym PBI is used, selected as the preferred polybenzimidazole because of its combination of thermal stability and processability.[4]

1.1. POLYBENZIMIDAZOLE

Polybenzimidazole (PBI), also known as poly[2,2'-(m-phenylene)-5,5'-bibenzimidazole], is a thermally stable, oxidatively resistant, heterocyclic polymer. It presents very high decomposition temperatures ($>500\text{ }^{\circ}\text{C}$), and does not show a melting point due to its lack of crystallinity. [3, 5]

Its molecular formula is $\text{C}_{20}\text{H}_{12}\text{N}_4$, with a physical appearance of golden brown dust, and a molar mass of n times 308.3 g/mol , where n is the number of monomers composing the polymer [6]. The structure of PBI is shown in the Figure below.

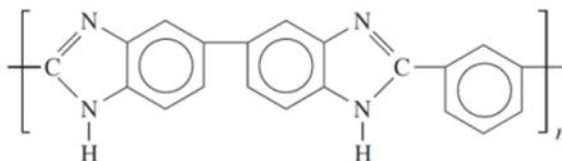


Figure 1. Poly[2,2'-(m-phenylene)-5,5'-bibenzimidazole] structure [14].

It is a basic polymer ($\text{pK}_a \sim 5.25$) and possesses both proton donor ($-\text{NH}-$) and proton acceptor ($-\text{N}=\text{C}-$) hydrogen bonding sites, which exhibit specific interaction with the polar solvents. However, fully aromatic PBIs are practically insoluble in most organic solvents due to the highly rigid aromatic polymer backbones and the strong interchain hydrogen bonding interactions, so only a limited number of highly polar aprotic organic solvents are capable of breaking these bonds and solubilize the polymer. The commonly used organic solvents include N,N-dimethylacetamide (DMAC), N,N-dimethylformamide (DMF), N-methyl-2-pyrrolidone (NMP), and dimethyl sulfoxide (DMSO). Among these solvents DMAC is the most widely used one [5,7,8,9].

When made into fibers, these show a retention of both strength and flexibility after exposure to flame. The stiff fibers also maintain their integrity when exposed to high heat and are mildew, abrasion and chemical resistant [10].

PBI presents extraordinary properties that position it above other polymers as a high-performance material, attracting many researchers to investigate PBI-based materials for applications with operating conditions well outside the typical realm of organic materials. These properties include extremely high glass transition and heat deflection temperatures, no melting temperature, and a very small % of elongation at break, among others, shown in Table 1.

Table 1. Polybenzimidazole properties

PROPERTIES	Units	Value
Melting point	°C	-
Heat deflection temperature	°C	427
Glass transition temperature	°C	425
Density (at 23 °C)	g/cm ³	1.3
Thermal conductivity (at 23 °C)	W/(K·m)	0.4
Tensile strength	MPa	130
Elongation at break	%	3
Young's modulus	MPa	5800

Although it is non-combustible, meaning the substance itself does not burn, if exposed to temperatures above decomposition temperature, it may decompose to produce corrosive and/or toxic fumes, such as carbon oxides, nitrogen oxides (NO_x), or other toxic gases.

SYNTHESIS

Industrial PBI synthesis is carried out by the polycondensation reaction of diacid and tetra-amine monomers or their derivatives. Depending on the particular nature of these initial reactants, the reaction can produce various PBIs, the general structure of which is shown in Figure 2 [5, 11].

This process must occur in a substantially oxygen free atmosphere to prevent oxidative degradation. An inert gas, such as nitrogen or argon can be passed continuously through the reaction zone during the polymerization, whether at room temperature or preheated to the reaction temperature [12]. The polymerization reaction is quite rapid, not needing either a pressure or vacuum cycle to obtain high molecular weight polybenzimidazole [13].

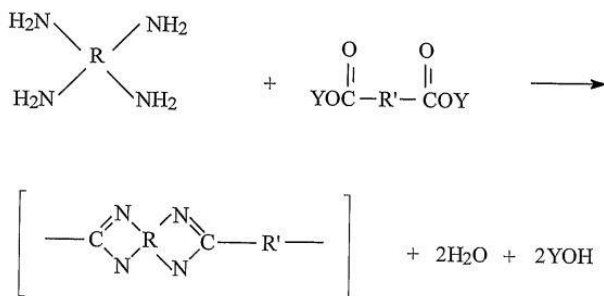


Figure 2. General PBI polycondensation [12]

As stated before, the only commercially available PBI, and the one that is most documented is poly[2,2'-(*m*-phenylene)-5,5'-bisbenzimidazole]. The industrial polymerization of this specific polybenzimidazole is conducted at temperatures above 200 °C, in the absence of solvent [8]. Most used reactants are 3,3',4,4'-tetraaminobiphenyl (TAB) and diphenyl isophthalate (DPIP). Figure 3 shows the mentioned reaction, as well as the structure of the species involved [9].

Performing the process in two stages has been studied, and finely pulverizing the intermediate product between these stages, favours the production of high molecular weight PBI [11, 12, 14].

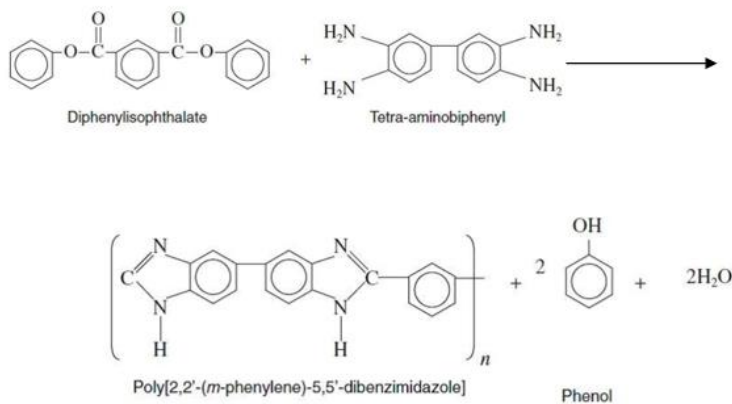


Figure 3. Solvent free PBI synthesis [9]

Although there are a few cases where catalyst is used, the majority of found examples do not, reason being that monomer purity, temperature, and removal of oxygen traces in the reactor are more important parameters than adding catalysts for obtaining high molecular weight polymer. [8]

A different method to synthesize PBI, commonly used on laboratory scale is homogeneous solution polymerization, where a solvent, usually polyphosphoric acid (PPA) is used to create a homogeneous mixture. This procedure takes place at lower temperatures (170 - 230 °C), and uses more stable initial monomers, such as isophthalic acid and TAB stabilized by tetrahydrochloride. However, reaction time drastically increases to times greater than 15h and, therefore, microwave techniques can be employed for the purpose of acceleration. [8]

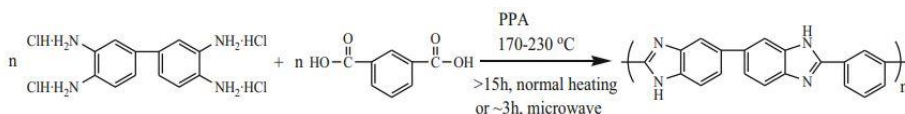


Figure 4. Homogenous solution PBI synthesis [8]

APPLICATIONS

As a high-performance polymer, polybenzimidazole has many applications. Initially, it was mainly used as fire-blocking, heat protection gear, and reverse osmosis membranes. However, as investigation progressed, its applications have multiplied to many others.

Although this project focuses on the production of PBI in powder form, it can be further processed in order to suit market needs. Other common forms include [15, 16, 17]:

- Granules/Pellets: Formed by compacting and shaping PBI powder. They provide convenience in handling and feeding into processing equipment such as injection molding machines or extruders.
- Films/Sheets: Thin materials with high thermal stability and chemical resistance, ideal for application in membrane electrode assemblies for fuel cells, flow batteries, and hydrogen pumps.

- Fibers: It is the main focus of industrial production. Exceptionally flame-resistant and strong, used in protective clothing and composites.
- Coatings: Applied onto substrates for protection against high temperatures and chemicals. They can be firmly adhered to steel, stainless steel, aluminum, copper, nickelchromium, glass, ceramics and plastics.
- Membranes: High-selectivity membranes for fuel cells, reverse osmosis, forward osmosis, nanofiltration, organic solvent nanofiltration, and gas separations

Therefore, considering all the possibilities it offers, PBI is present in many industries, some of the main ones are [17, 18, 19, 20]:

a) Aerospace:

PBI is used in aerospace applications for components requiring high temperature resistance and mechanical strength. This includes aircraft engine parts, thermal insulation, seals, gaskets, and composite materials. They can be used for tactical missile structures, high speed projectiles, aircraft brake assemblies and radiation-resistant structures, among others.

b) Automotive:

In the automotive industry, PBI is employed in high-temperature components such as gaskets, seals, connectors, and insulating materials for engines, exhaust systems, and electrical systems.

c) Chemical Processing:

PBI's resistance to a wide range of chemicals makes it valuable in chemical processing industries. It is used in membranes, pump components, and other equipment requiring resistance to aggressive chemicals, high temperatures, and pressures.

d) Electronics:

PBI is used in electronics for applications requiring high thermal stability and electrical insulation. It is employed in components such as insulators, connectors, circuit boards, and protective coatings for electronic devices operating in high-temperature environments.

e) Oil & Gas:

In the oil and gas industry, PBI is utilized in equipment and components exposed to high temperatures, aggressive chemicals, and harsh environments. This includes seals, gaskets, packings, valve seats, and insulation materials for downhole tools, pumps, and pipelines.

f) Military and Defense:

PBI's flame resistance and thermal stability make it suitable for military and defense applications. It is used in protective clothing, equipment components, and insulation materials for military vehicles and aircraft.

g) Textiles:

PBI fibers are employed in high-performance textiles for applications requiring flame resistance, thermal stability, and chemical resistance. This includes aluminized crash rescue gear, aircraft fire blocking layers and wall fabrics, and protective apparel for firefighters, military personnel, industrial workers and racing car drivers.

h) Other applications include asbestos replacement in seals, abrasives and brake pads, as well as bonding diamond abrasive cutting tools and wires.

1.2. MARKET STUDY

PBI was first introduced in 1959 and further developed by Marvel and Vogel in 1961, opening the field of high temperature polymers. This was followed by AFML and NASA funded programs for basic studies and for structural and textile applications of polybenzimidazoles to meet new aircraft and aerospace material needs.

The company Celanese Corporation began manufacturing PBI under the brand name "Celazole" in the late 1960s, but it was not made commercially available until 1983.

Throughout the 1970s and 1980s, PBI started to find practical applications in various industries, such as aerospace, automotive, electronics, and protective clothing sectors, gaining a distinct profile and reputation in the fire retardant materials industry.

In 2005, Celanese Corporation sold the PBI fiber and polymer business to PBI Performance Products Inc. Since then, as the benefits and applications of polybenzimidazole became more known, other companies entered the market to meet the growing demand. [4, 10]

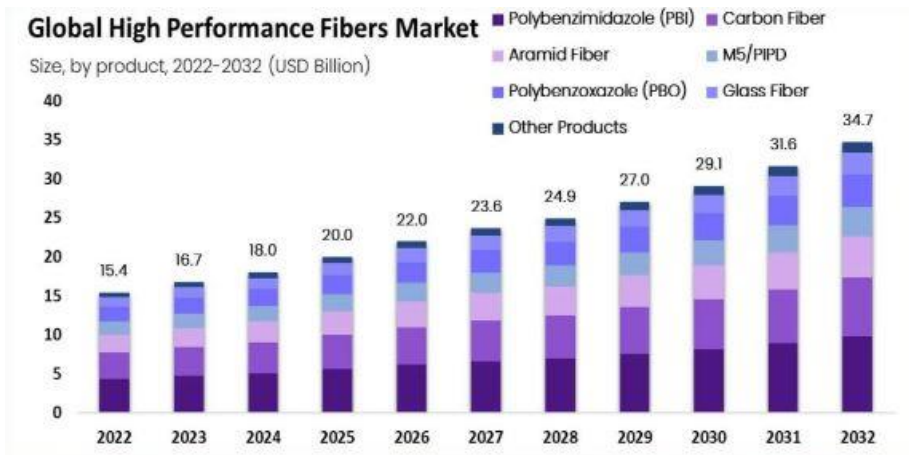


Figure 5. Global High Performance Fibers Market Forecast [20]

There is little public information about the market size of polybenzimidazole, however, some can be found regarding PBI fiber. As stated previously, the main commercial outcome for polybenzimidazole is producing fibers with it, therefore, in order to get an idea of the production scope of PBI, the high performance fibers market must be studied.

The Global High-Performance Fiber Market size reported in 2022 was USD 15.4 billion and is projected to reach USD 34.7 billion. In 2022, PBI accounted for the largest revenue share in High-Performance Fibers Market, even above carbon fiber, and is expected to continue to do so [20]. This forecast is shown in Figure 5.

In 2019, the polybenzimidazole fiber market size was valued at USD 1.2 Billion, and it is projected to increase in the following years, due to the growing demand for high-performance materials, reaching about USD 2.5 Billion by the end of 2030 [19]. This estimate is illustrated in Figure 6.

Yearly production values are not posted to the public, however, considering the wide price range of PBI, that varies from 2 - 200 USD/kg [21], some orientative production figures can be calculated. For the sake of conducting this project, an estimate value of 100 000 tonnes/year will be selected as annual global production.

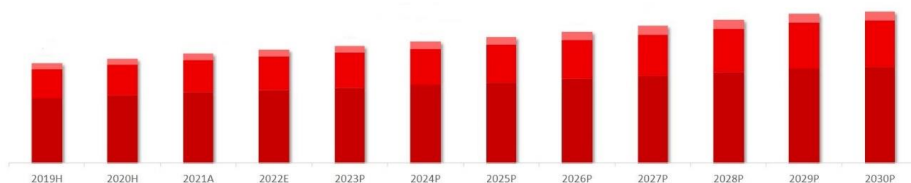


Figure 6. Global PBI Fiber Market Forecast [19]

The majority of PBI production is carried out in the United States, followed by Europe, China and Japan. Its main manufacturers are the following companies [19, 22, 24]:

- PBI Performance Products
- Celanese Corporation
- Atkins & Pearce
- Mitsubishi Chemicals
- Goodfellow
- Solvay SA
- Polymics, Ltd.
- Bally Ribbon Mills (BRM)
- TenCate Protective Fabric

2. OBJECTIVES

Given the rising demand for high-performance materials and recognizing the significance of polybenzimidazole (PBI) within this sector, this project aims to develop a basic engineering design for a Polybenzimidazole production plant. This facility has an annual capacity of 1000 tonnes, representing 1 % of the global annual production. Batch size is 5 tonnes.

To accomplish this objective, 3,3',4,4'-tetraminobiphenyl (TAB) and diphenyl isophthalate (DPIP) will be used as raw materials to obtain a polymer with an average chain molecular weight of 30,000 Mw, a purity of 99 % and a particle size between 100 and 200 μm . As a final product, PBI will be packaged in powder form in big bags of 500 kg. The plant will operate in discontinuous with the aim of increasing flexibility and favouring the multifunctional use of equipment.

To achieve the main objective, the first action that is accomplished is the market research, that helps contextualize the study and determine production scope. Next, a detailed patent analysis will be carried out, in order to select the most appropriate method for the production of the polymer and retrieve the necessary data.

Once the production process is determined, the research will proceed with the detailed development of a process diagram, mass balance, and the definition of the process recipe to establish the necessary steps and equipment required in production. Following this, the design phase will involve equipment selection and specification, prioritizing key parameters to ensure optimal performance and operating conditions. A Process and Instrumentation Diagram (PI&D) is then created.

Finally, A time study will be conducted, determining batch time (BT) and cycle time (CT). A detailed production planning will be developed through a scheduling strategy and the calculation of the Key Performance Indicator (KPI's) that give information on the level of production, ultimately ensuring a robust and efficient PBI production process.

3. PATENT RESEARCH

The intent of this study is to carry out the basic design for the production and conditioning of polybenzimidazole through the polycondensation reaction of tetra-amine and dicarboxylic acid monomers or their derivatives. To better pursue this objective, research of patented processes is necessary, in order to analyse the already existing production methods and extract useful data from them.

This research should provide the necessary information to choose the procedure that will be followed in this particular study. From all the considered patents, shown in Table 2, the most appropriate one will be elected as a base for the PBI plant design, while taking into consideration technical and economic factors.

In this case, patent CA2612722A1 has been chosen, reason being that it is the most complete one that describes an industry big-scale process, while the others mainly focus on laboratory scale. Therefore, knowing that scaling-up can be complex, it is more convenient to avoid it if possible, or at least to minimize it, by taking the most similar sized description as a guide.

This patent provides a description of a solvent-free two-step polycondensation process, ensuring the production of high molecular weight PBI. It uses no catalyst and, like the rest, happens at atmospheric pressure and uses nitrogen to provide an oxygen-free environment. It is noteworthy that the applicant of this patent is the main producer of polybenzimidazole, PBI Performance Products, Inc.

However, information from the other listed patents is also used, since they provide details not mentioned on CA2612722A1, such as the use of distillation for the removal of reaction byproducts. Moreover, it can be observed that while the polymerization is described, the conditioning operations are not. This is why other sources, such as articles or books, have been additionally made use of. The process will be further described in the following section.

Table 2. Patent reasearch

PATENT	OPERATIONS	CATALYST	CONDITIONS	TIME (h)
CA2612722A1	Polycondensation 1	-	290 °C	1.5
	Agitation termination		290 °C	0.75
	Cooling		25 °C	-
	Prepolymer pulverizing		-	-
	Polycondensation 2		380 °C	2
US4452971A	Polymerization + Distillation	Tin-containing catalyst (eg. stannic chloride, dibutyltin oxide)	400 °C	2
	Cooling	-	25 °C	-
US2006004182A1	Polycondensation 1	-	290 °C	-
	Agitation termination		290 °C	1.25
	Cooling		25 °C	-
	Prepolymer pulverizing		-	-
	Polycondensation 2		330 °C	4
CN110592712A	Solvent heating	-	80 °C	1.5
	Polycondensation 1		150 °C	2
	Polycondensation 2		210 °C	10
US2895948A	Diamine preparation	Raney nickel catalyst	-	-
	Polycondensation	-	250 °C	4

4. PRODUCTION PROCESS

From the market research, an estimate of 100 000 tonnes/year is drawn for global PBI production. Based on this, the selected production for the designed plant represents 1 % of the global one, being 1000 tonnes/year. The obtained polymer will have an average chain molecular weight of 30 000 Mw, a purity of 99 % and a particle size between 100 and 200 μm . These standards are set to match those offered by other manufacturers.

The basic plant design carried out in this project includes the production and conditioning processes of PBI, as well as packaging. Therefore, that is what is inside the battery limit, excluding subsequently plant services, raw materials storage and the treatment of all generated waste.

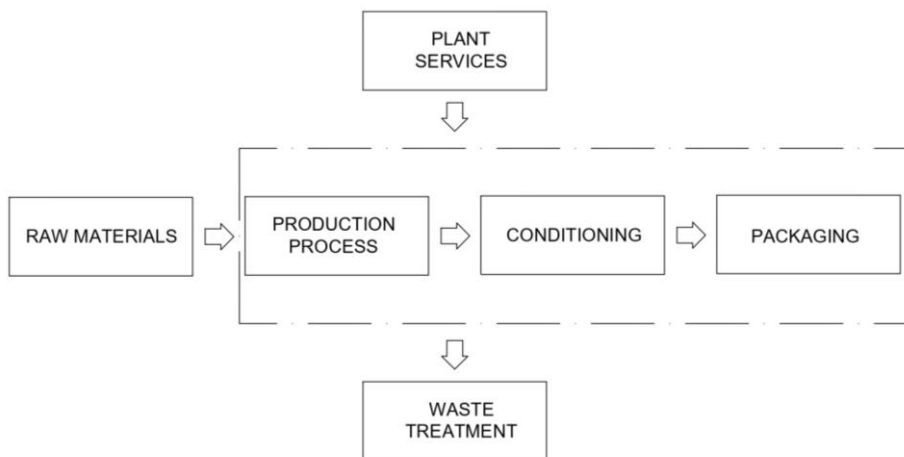


Figure 7. Battery limits

4.1. RAW MATERIALS

As has been established, the monomers used as reactants are 3,3',4,4'-tetraminobiphenyl (TAB) and diphenyl isophthalate (DPIP). TAB is a red-to-brown hygroscopic solid in powder form, obtained through multi-step organic synthesis processes, typically involving the nitration and reduction of biphenyl. DPIP consists of a white to off-white solid powder derived from the esterification of isophthalic acid with phenol.

Providing safety in each section of the plant is essential, therefore, the properties of the raw materials must be understood in order to assure a correct handling, as well as proper working specifications of the equipment. TAB is combustible and it is classified according to Regulation (EC) No 1272/2008: Acute toxicity, Oral (Category 4), H302; Eye irritation (Category 2), H319; Germ cell mutagenicity (Category 2), H341; Carcinogenicity (Category 1B), H350. On the other hand, DPIP is not classified as a hazardous substance, and is non-combustible.

In addition to TAB being combustible, the temperature at which the reaction takes place, 300 °C, is superior to the flash points of both monomers, making it imperative to inertize the equipment before the operation takes place. Due to the lack of information about the precise properties of the formed prepolymer, inertization will be carried out in all equipment where the reaction is not complete, that is to say V-01, M-01 and V-02. For the rest, it will not be necessary, given that PBI is stable in the presence of oxygen.

The solvent used in the purification stage is dimethylacetamide (DMAC), which has a density of 940 kg/m³ and a boiling point at atmospheric pressure of 160 °C. Nevertheless, it is also necessary to study the variation of the boiling point at different pressures, which is shown in Figure.

Table 3. Raw materials properties [24, 25, 26]

PROPERTIES	UNITS	TAB	DPIP
Phase (25 °C)	-	Solid	Solid
Colour	-	Red to brown	White
Melting point	°C	175-177	136-138
Boiling point	°C	344	499
Flash point	°C	280	223
Density	g/cm ³	1.3	1.2
Water solubility	-	Very poor	Insoluble
Molar weight	g/mol	214.27	318.32

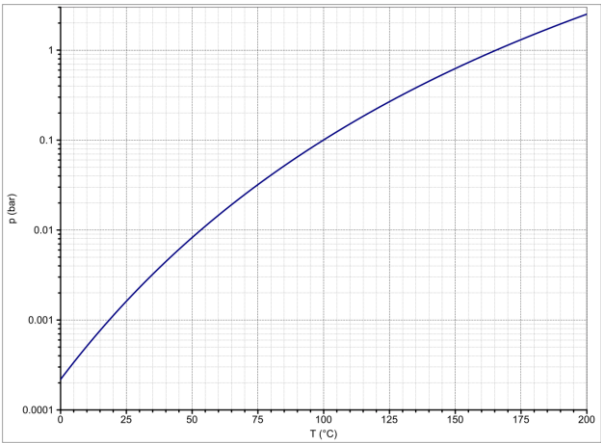


Figure 8. Boiling point DMAC vs. Temperature [27].

4.2. BLOCK DIAGRAM

The block flow diagram (Figure 8) provides a broader overview of the process, clearly illustrating the various operations involved in the overall synthesis of polybenzimidazole.

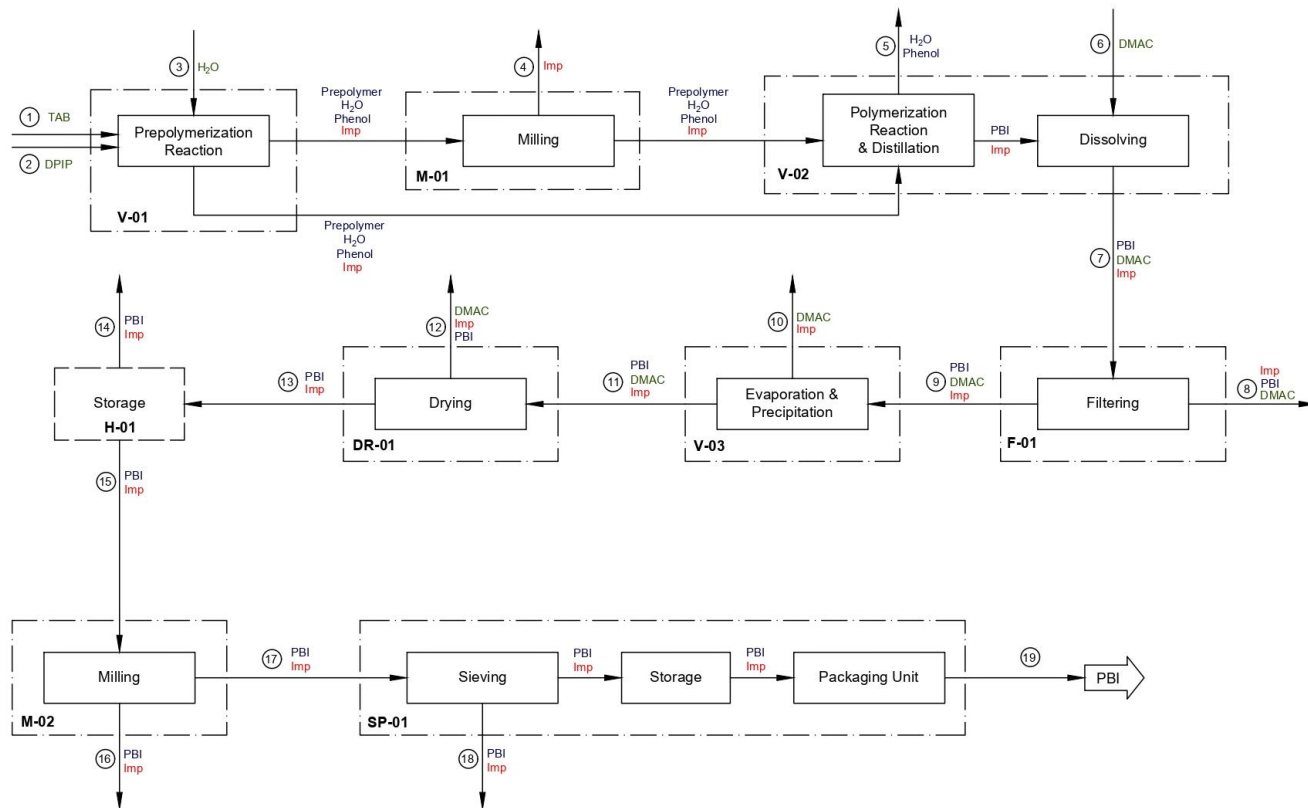


Figure 9. Block diagram plant

4.3. MASS BALANCE

A mass balance must be carried out for the determination of raw materials necessary for the obtaining of the desired amount of product, as well as the dimensioning of equipment and to help establish occupation times. The block diagram used in Figure 8 illustrates the details of the process and numerates the streams that must be defined.

The batch size of the designed plant is fixed at 5 tonnes/batch of PBI, with a 99 % purity. The mass balance is carried out, as shown on Table 5, and shows 3805 kg of TAB and 5647 kg of DPIP are necessary to make possible the desired production size.

To simplify the mass balance analysis, a few assumptions must be made, which are the following:

- The reaction is complete: The polycondensation reaction taking place in vessels V-01 and V-02 is irreversible and complete, neglecting the possibility of lateral depolymerisation, therefore, all TAB and DPIP react. This is backed by the fact that in V-02, a distillation takes place, constantly removing water and phenol produced as by-products, and shifting the direction of the reaction completely to the right.
- In vessel V-01, the first discharge represents the 95 % of the contents of the tank. The remaining 5 % is pressure washed with 2000 L of water, resulting in a wash mixture that is directly introduced in V-02.
- All water and phenol are removed by the forementioned distillation before proceeding to the next steps. This includes the water and phenol produced in the polycondensation, as well as the wash water.
- The filter removes a total of 150 kg of solid impurities, which retain a 10 % of humidity in weight from the PBI dissolution in DMAC.
- In the evaporation process, the vaporized DMAC drags a total of 25 kg of liquid impurities. The remaining concentrate is wet PBI, with a humidity of 20 % in weight of DMAC.
- The drying is complete, meaning the PBI that exits the dryer DR-01 retains no humidity, all DMAC is removed.

- In the sieve from SP-01, all losses are due to overgrinding, no coarser particles are obtained. This is because in the ball mill M-02, only particles of the desired size (or smaller) are allowed to exit.

All equipment losses are shown in Table 4.

Table 4. Equipment losses	
Equipment	Losses [%]
M-01	1.0
DR-01	0.5
H-01	0.5
M-02	0.5
SP-01	2.0

All of these are initial assumptions, and although they are funded and are expected to be reflected in reality, they are only an approximate estimate. Therefore, once the process plant is operational, they must all be confirmed.

Table 5. Mass balance

(kg)	In	In	In	Out	Out	In	Tr	Out	Tr	Out	Tr	Out	Tr	Out	Tr	Out	Tr	Out	Out
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19
TAB	3805	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
DPIP	-	5647	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Water	-	-	2000	-	2639	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Phenol	-	-	-	-	3339	-	-	-	-	-	-	-	-	-	-	-	-	-	-
PBI	-	-	-	-	-	-	5157	4	5153	-	5153	26	5128	26	5102	51	5051	101	4950
DMAC	-	-	-	-	-	15472	15472	11	15460	14419	1041	1041	-	-	-	-	-	-	-
Impurities	-	-	-	-	-	-	227	-	77	-	52	~0	52	~0	52	1	51	1	50
Imp I (s)	-	-	-	90	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Imp II (s)	-	-	-	-	-	-	-	150	-	-	-	-	-	-	-	-	-	-	-
Imp III (l)	-	-	-	-	-	-	-	-	-	25	-	-	-	-	-	-	-	-	-
TOTAL	3805	5647	2000	90	5978	15472	20856	165	20690	14444	6246	1067	5180	26	5154	52	5102	102	5000

4.4. RECIPE

This section offers insight into the polybenzimidazole (PBI) production process, as designed in this study. It encompasses each stage included of the operation, except those outside the battery limit, therefore including synthesis, purification and packaging of the product. Most of the stages are designed to operate discontinuously, except a few that will be pointed out.

Before starting, a few things should be highlighted. For each equipment involved, a final cleaning stage must be considered after usage. It will be adapted to the individual specifications of the unit, ensuring a correct removal of any leftover materials and contaminants that could affect the quality of future operations.

On top of that, it is imperative that those vessels where the reaction takes place, or used while the reaction has not completed (V-01, V-02 and M-01) are inerted beforehand, due to the risk of oxygen presence influencing the reaction, as well as a general safety measure.

Quality controls will also be carried out at different stages of the process, making sure the obtained product meets all quality standards.

The production plant is overall based on a zero wait (ZW) operating philosophy, where the intermediate products are transferred to the succeeding unit as soon as the current task finishes. However, a few units follow a finite intermediate storage (FIS) philosophy, due to the fact that there is a waiting period because of the timing of some operations.

The synthesis of polybenzimidazole consists of a two-step solvent free polycondensation reaction with an intermediate pulverization. This is followed by a purification through dissolution and filtering, with the solvent being removed afterwards by drying the product. Finally, the particle size of the resulting powder is corrected to the desired one and packed in big bags. Each of these are described in more detail below.

4.4.1. Raw materials reception

The process begins with the reception of 3,3',4,4'-tetraaminobiphenyl (TAB) and diphenyl isophthalate (DPIP) at the plant. They will both be purchased in liquid form, heated to 200 °C and, therefore, they will be stored in heat-insulated tanks.

Although the storage temperature is below both of their flash points, a correct handling and storage is critical, especially in the case of TAB, which is classified as hazardous and combustible.

4.4.2. Polycondensation reaction

The polybenzimidazole (PBI) synthesis begins with starts with the sequential introduction of the key components into vessel V-01. This is, however, preceded by the inertization discussed above, where the vessel is degassed and filled with nitrogen. Then, 3805 kg of 3,3',4,4'-tetraminobiphenyl are introduced through a pipeline by a pump. At the same time, mixing and heating are started, until achieving a temperature of 300 °C. Next, 5647 kg of diphenyl isophthalate are loaded also through a pipeline by a pump, while continuing mixing and heating, ensuring an homogenous mixing of the monomers and a stable temperature at 300 °C.

At that point, the first step of the reaction begins, which takes place at 300 °C, at atmospheric pressure, for a duration of 1.50 h. After that, mixing is terminated and heating is continued allowing the mass to bubble into a friable mass, for an additional holding time of 45 minutes, after which the contents are cooled to room temperature. As a result of the polycondensation, a solid oligomeric PBI prepolymer is obtained, as well as the by-products water and phenol. The presence of said by-products is what causes the prepolymer to take the form of a friable foam. [12]

Agitation is activated again to partially break down said foam, which will be discharged into the humid mill M-01 through a pipeline by a positive displacement pump in order to be finely pulverized. Although before discharge, a quality control is carried out, making sure the process is advancing as designed. The mill functions continuously, its input being the discharge of vessel V-01, and its output charging vessel V-02.

When this first discharge is complete, the content that remains in the vessel is washed by high pressure water jets, using 2000 L of water. Then, there is a second discharge, of the prepolymer wash mixture, which is directly introduced into the vessel V-02 through a pipeline by a pump. Vessel V-02, as well as M-01 are also inerted prior to their usage, as mentioned previously.

The second step of the reaction proceeds in solid state in vessel V-02. As it receives the first charge, of the pulverized prepolymer, agitation and heating starts. After the second charge,

of the prepolymer wash mixture, heating and agitation continue, and the polymer molecular weight is advanced until the polymerization is complete, producing PBI of approximately 30 000 kg/kmol. This is achieved because vessel V-01, as well as acting as a reactor, operates as a simple distiller, removing therefore the by-products and shifting the reaction equilibrium toward the formation of PBI according to Le Chatelier's principle. The reaction and distillation occurs at 380 °C, at atmospheric pressure, for a duration of 2 h.

Afterwards, the polymer is cooled down to a temperature of 100 °C. At this point, another quality control is carried out, to analyse the produced PBI and assure it satisfies all standards.

4.4.3. Filtering

The polybenzimidazole produced in the previous section contains many impurities, which need to be removed before it is able to meet the specifications of the final product. The selected route to do so is to dissolve it in a suitable solvent, and separate the insoluble impurities from it through a filtration. In this case, the most adequate solvent is dimethylacetamide (DMAC).

The dissolution occurs in vessel V-02, after PBI has been obtained, DMAC is charged into the tank through the pipeline and a pump and with the help of agitation, the polymer dissolves. The solution is 25 % of polybenzimidazole and 75 % DMAC, in weight.

Half of the solution is charged through the pipeline and a pump into the bag filter F-01, which operates continuously, vertically and has a bag capacity that makes it necessary to operate the filtration in two sets. F-01 discharges inside of the vessel V-03 through the pipeline and a pump. After half of the solution is filtered, the bag in the filter needs to be changed, which will be done by the workers with the help of a bridge crane situated on top of F-01. A new bag is introduced and the filter is conditioned for the other half of the solution. The same procedure is repeated again, in the end, a total of 150 kg of solid impurities are removed, with an additional humidity content of 10 %.

This leaves the purified dissolution in V-03, ready for evaporation.

4.4.4. Drying

To obtain PBI as specified, in powder form, the solvent must be eliminated through two steps. The first is evaporation, which leads to a polymer paste of 20 % humidity, the second one is a drying that removes all humidity and leaves the PBI completely dry.

As has been explained, the evaporator V-03 is charged twice from F-01, after this, evaporation and precipitation begin, at atmospheric pressure, and at 180 °C °C. When complete, a quality control takes place to check some parameters, such as humidity level, and assure that they meet the set expectations.

The second step happens in the dryer DR-01, which has a working capacity of about half the volume that must be treated. Therefore, it operates twice per batch, at a vacuum of 0.01 bar to allow a lower boiling point, thus it operates at 60 °C, and is charged from V-03 through a pipeline by a positive displacement pump.

Hopper H-01 is located underneath the dryer DR-01, thus, the dryer is discharged by gravity into the hopper. At this point, the PBI is completely dry and the only thing that separates it from the final product is a consistent and correct particle size. For that reason, once the hopper has been filled, the most complete quality control is carried out, ensuring all other specifications of the PBI meet quality standards.

4.4.5. Milling & packaging

As stated above, the only thing that remains is correcting the particle size of the polybenzimidazole. This is done in ball mill M-02, which is charged from H-01 through an Archimedes' screw. After this, the PBI enters SP-01, which consists of a sieve, a hopper and a packaging system, arranged vertically, so each of them can discharge by gravity to the following equipment.

The mill, as well as the sieve, function continuously and are configured in series to work simultaneously. The sieve contains two decks, one of 200 µm, and one below of 90 µm. In this way, any coarse or fine particles are removed from the final product, which must be from 100 to 200 µm. The output from the sieve is charged into the hopper situated underneath it, which will

hold the product until milling and sieving is complete. Before proceeding, one last quality control is performed, this time to assure a correct particle size distribution. In the cleaning stage, the mass retained by the sieve (PBI out of specification) is removed.

The final step is packaging, through a packaging system that will fill 10 big bags of 500 kg. These big bags will be moved by workers with the help of the necessary transportation machinery, and lastly they will be labelled, indicating all specifications of the product.

5. EQUIPMENT SPECIFICATION

A crucial part of plant design is selection or design of the process equipment. This selection must consider at all times the specific process and particular task assigned to them. Thus, the chemical and physical properties of the present species, as well as the quantities that are treated, and occurring reactions are critical for this design.

Many times in the industry, however, instead of designing the units, it is more practical to access existing equipment, which tends to be standardized, avoiding the higher costs that the manufacture of highly specialized equipment usually imply, enhancing efficiency and cost-effectiveness in large-scale production.

Therefore, in this study, the vessels V-01, V-02, V-03 and the hopper H-01, as well as the hopper from the SP-01 complex are specifically designed, while the mills M-01, M-02, the filter F-01, the dryer DR-01 and the sieve and packaging unit in SP-01 will be obtained from commercial catalogues provided by various companies.

In this section, a basic specification is conducted, with the necessary guides [32, 36, 37]. The specification datasheets of each piece of equipment are provided in Appendix 1.

5.1. REACTOR VESSEL (V-01)

In vessel V-01 the polycondensation reaction of TAB and DIP occurs, producing a low weight oligomeric prepolymer precursor of PBI, as well as water and phenol. This process involves the polycondensation reaction and, due to the presence of water and phenol, the formation of a friable foam.

Vessel volume is determined considering volume of reactants and keeping in mind that the formation of foam will increase the volume of the product considerably. Therefore, to guarantee sufficient room for it, as well as a headspace of 10-20 %, the volume of the vessel is 15 m³.

The geometry of the vessel is cylindrical with a Kloppe head, and the selected material is 316 L stainless steel. The height/diameter ratio (L/D) is set at 2, therefore, a total height of 4.9 m and a diameter of 2.1 m is obtained, with a wall thickness of 9 mm.

The reactor operates at atmospheric pressure, however, an increase of pressure with advance of reaction due to gas production (water and phenol) is expected. Design pressure is usually set between 10 % and 20 %. The relief valve is set at design pressure, which is connected to a pressure transmitter.

In terms of agitation, due to the increase of viscosity throughout the advance of the reaction, an impeller composed of both an anchor and a helical ribbon is chosen (Figure 10). The agitator has a diameter (D_a) of 1.8 m and a tank bottom separation (C) of 0.7 m. The mixer is equipped with a motor with variable velocity, increasing torque as the reaction advances.



Figure 10. Anchor and helical ribbon agitator.

The polycondensation occurs at 300 °C and, in addition, the prepolymer is afterwards cooled down to room temperature. Although heat exchange is not designed in this study, the vessel is designed with a half pipe jacket with a surface contact area of 33.6 m² that works with thermal oil.

The cleaning of the vessel is done with pressure jet mouths, due to the presence of the friable foam, which is otherwise difficult to eliminate.

Other relevant information are the connections of the vessel, which include the inlet for the raw materials, the outlet, connected to a positive displacement pump. There must also be an

inlet for nitrogen and a manhole. In the half pipe jacket, there is also an inlet and an outlet for the thermal oil.

Finally, although not detailed, a temperature control loop is in place, connected to the oil supply, as well as level alarms and a pressure transmitter connected to the relief valve. This situation is the same for vessels V-02 and V-03.

5.2. HUMID MILL (M-01)

The total mass treated by the humid mill M-01 is approximately 9 tonnes, containing an oligomeric precursor PBI in the form of a friable foam with water and phenol trapped in it, which must be finely pulverized before introducing it in the second reactor vessel V-02.

The mill is supplied by SOLDO Cavitators [28, 29]. It works continuously with a mass flow of 4 tonnes/h, therefore the operation has a duration of 2 h, reaching a particle size of about 1 μm . It is constructed of 316L stainless steel and works at a pressure of 1.5 bar and a temperature of about 25 °C.



Figure 11. Humid mill [33]

5.3. REACTOR VESSEL (V-02)

The second reactor, where the polymerization reaction is completed, consists of a vessel, similar to V-01. In V-02, the polymerization reaction is complete, as well as a simple distillation.

Vessel volume is defined by the maximum occupancy, which occurs at the end of the process, when the PBI is dissolved in DMAC. Considering their volume and a headspace of 10 – 20 %, the volume of the vessel is 25 m³.

The geometry of the vessel is also cylindrical with a Kloppe head, and the selected material is 316L stainless steel. The height/diameter ratio (L/D) is set at 2, therefore, a total height of 5.8 m and a diameter of 2.4 m is obtained, with a wall thickness of 9 mm.

The reactor operates at atmospheric pressure, and the phenol and water vapor are removed through a simple distillation and conveyed to a condenser.

In terms of agitation, considering the reaction in place is in solid phase, the same kind of mixer as vessel V-01 is chosen (Figure 10), also with a motor of variable velocity, since it will need less when performing the dissolution. The agitator has a blade diameter (D_a) of 2.4 m and a tank bottom separation (C) of 0.8 m.

The polycondensation occurs at 380 °C and, in addition, the polymer is afterwards cooled down to a temperature of 100 °C. The vessel is designed with a half pipe jacket with a surface contact area of 47.6 m² that works with thermal oil.

Other relevant information are the connections of the vessel, which include the inlet for the prepolymer and another for the DMAC, the distillate outlet and the PBI solution outlet, connected to a pump. There must also be an inlet for nitrogen and a manhole. In the half pipe jacket, there is also an inlet and an outlet for the thermal oil.

5.4. BAG FILTER (F-01)

The filter has the task of separating the impurities of the PBI solution, in order to purify the product. In total, the solid impurities removed are 150 kg.



Figure 12. Bag filter [30]

The bag filter is supplied by Peiró [30]. The bag has a diameter of 180 mm and a length of 810 mm. With these dimensions, the filter only has the capacity to remove half of the impurities at a time, filling $\frac{3}{4}$ of the bag, and for this reason, it must operate twice. The bags are changed manually with the help of a bridge crane.

It works continuously at a flow rate of around 20 m³/h, at a temperature of 100 °C and atmospheric pressure. It has a length of 1 m.

5.5. EVAPORATOR (V-03)

In vessel V-03, evaporation and precipitation take place. Thus, the primary goals are to ensure a correct mass transfer and heat transfer, both of which will improve the rate of evaporation.

It operates at atmospheric pressure, and at a temperature of 180 °C, above DMAC boiling point.

As for the vessel design, it is exactly the same as vessel V-02, the only thing that changes are the uses of the mouths. Here, the mouths that differ are those used for the PBI solution inlet, and for the concentrate outlet, connected to a positive displacement pump. The rest of the setup is the same.

5.6. DRYER (DR-01)

The vacuum dryer is used to remove the remaining humidity of 20 % on the PBI. This operation is performed at a pressure of 0.01 bar, and, therefore, the boiling point of DMAC is lower, allowing the dryer to function at a temperature of 60 °C.

The dryer is supplied by Paul O. Abbe [31]. It is a horizontal axis rotary vacuum dryer with a total volume of 5.9 m³ and a maximum working capacity of 3.9 m³, equivalent to 65 % of the total volume.

It has a double cone with a diameter of 2.30 m and a height of 3.58 m that provides unobstructed tumbling of solids. It has a rotation speed of 3 rpm and it is equipped with a jacketed surface area of 16.17 m². It does not need to be connected to the plant service, since the equipment unit is connected to its own vacuum system.

Pressure and temperature will be monitored by the means of both pressure and temperature indicators.

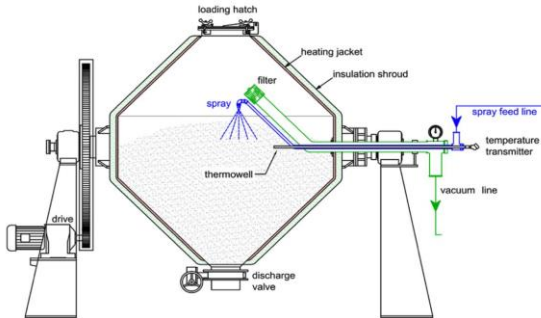


Figure 13. Vacuum dryer [31].

5.7. HOPPER (H-01)

The hopper must hold approximately 5200 kg of PBI, as shown in the mass balance, which, with a density of 1300 kg/m^3 , has a volume of around 4 m^3 . The design of the hopper is conducted with the assumption that 80 % of the volume is used for storage, so the total volume of H-01 is set to 5 m^3 , with a height/diameter ratio of 2:1 to facilitate gravity discharge.

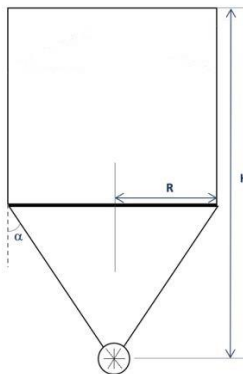


Figure 14. Hopper configuration

The hopper (Figure 14) has a geometry composed by a cylindric part and a conical one on the bottom where it discharges from through a rotatory valve. The height-diameter ratio of the cylindrical part, as well as the angle of the conical section, that goes from the diameter of the cylinder to the diameter of the outlet. The latter is set to 30° [32], which in turn gives a diameter of 1.6 m and a height of 3.2 m.

The outlet of the hopper is connected to a rotary valve, in order to minimize gas transference and facilitate the powder discharge. In order to monitor the level of the powder, a lever sensor will be installed in the hopper, connected to an alarm.

Depending on the interaction between the stored product and the wall, the gravity discharge might need assistance from a vibration system to avoid the hopper emptying only partially, caused by funnel flow.

5.8. BALL MILL (M-02)

The ball mill is supplied by Henan Baichy Machinery Equipment [33].

The total mass treated by the mill M-02 is approximately 5 tonnes. It works continuously with a mass flow of 2 tonnes/h, therefore the operation has a duration of 3 h, reaching a particle size between 100 and 200 μm . It is constructed of 316L stainless steel and works at atmospheric pressure and a temperature of about 25°C .



Figure 15. Ball mill [33]

5.9. SIEVE AND PACKAGING (SP-01)

The sieve and packaging equipment are designed as one block, SP-01. Between the sieve and the packaging, there is a hopper, since the packaging unit operates particularly. The hopper has the exact same design as H-01.

a) Sieve:

It operates continuously and simultaneously to the ball mill M-02. Thus, it adapts to the flow rate of the mill. It has a total diameter of 2 m and an effective screening area of 2.9 m².

The sieve is supplied by Eversun Machinery [34]. It operates at atmospheric pressure and at a temperature of 25 °C and it is constructed with 316L stainless steel. The configuration is of two decks, one on top of 200 µm and one below of 90 µm. Therefore, the PBI of the desired particle size is obtained, while removing any fine or coarse particulates.

The packaging unit consists of a big bag filler, it is supplied by Palamatic Process [35]. It operates at atmospheric pressure and at a temperature of 25 °C, and is mainly constructed of 316L stainless steel.

It is used to fill 10 big bags of 500 kg each. The device has a height of 4 m, a weighing precision of ± 500 g and a installed power of 1.7 kW.



Figure 16. Sieve [34]



Figure 17. Packaging unit [35]

5.10. PLANT SERVICES

Design of plant services are outside battery, as has been established. Nevertheless, it is important to briefly state the necessary services.

- a) Nitrogen: An inert gas supply is necessary to inertize the equipment that requires it.
- b) Deionized water is needed for equipment cleaning.
- b) Thermal monofluid: For all heat exchange operations, a monofluid system is essential.

In the case of this study, a thermal oil is used, which is heated to 420 °C in a boiler, operating with water steam.

In the case of refrigeration, the oil is cooled to 50 °C in one case and to 10 °C in the other, in a heat exchanger working with glycol water at -10 °C.

The thermal oil at 420 °C is used for heating of vessels V-01, V-02 and V-03. For cooling of vessel V-01, the thermal oil at 10 °C is used. Finally, for cooling of vessel V-02, the oil at 50 °C is employed.

5.11. ADDITIONAL EQUIPMENT

The following additional equipment is required for the plant production:

- Centrifugal pump (x2): Used to convey fluids of low viscosity ($\sim 1000 \text{ kg/m}^3$) from one equipment to the next, as specified in the recipe.
- Positive displacement pump (x2): Used to convey fluids of higher viscosity from one equipment to the next, as specified in the recipe.
- Rotatory valve (x2): Used for the powder discharge from the hoppers, to minimize gas transfer.
- Archimedes' screw: Used to transport powder upwards from the ball mill M-02 to the hopper H-01.

6. P&ID DIAGRAM

7. PRODUCTION PLANNING

This section is dedicated to the structuring of the overall annual batch production. To do so, it is necessary to study in detail the timing of a single batch production and, once this is complete, to consider various alternatives for yearly campaign organisation.

This analysis is essential, since the selection of the optimal campaign arrangement implies a more efficient management of the plant, which can mean higher economic benefits without the need for large financial investments.

7.1. TIME STUDY

The first step is to conduct a detailed time study of all operations conducted at the plant, shown in Table x. This is done by listing all tasks carried out in each unit and setting a time for all of them, considering most equipment functions discontinuously, but there are a few exceptions which function discontinuously, M-01, F-01, M-02 and the sieve in SP-01.

Another consideration is that, as stated previously, the plant follows a Zero Wait Operational Philosophy, when it is possible. When not, it follows a Finite Intermediate Storage Philosophy.

Furthermore, there are two units, the filter F-01 and the dryer DR-01, that operate in two cycles. In the case of the filter, it is because the bag must be changed after the first cycle. For the dryer, it is due to its dimensions, since it can only process half of the PBI at a time.

Finally, although it is not explicitly indicated in Table x, the time set for packaging in SP-01 includes the big bag filling time (15 min x10) and the time for the big bag to be moved and replaced for an empty one (15 min x10).

For better comprehension, operations that occur simultaneously in Table x have been highlighted with the same colour.

Table 6. Occupation times study

UNIT	OPERATION	OT [h]	Start t [h]	End t [h]
V-01	Inertize	0.5	0	0.5
	Charge of TAB (mixing and heating)	0.5	0.5	1.0
	Heating to 300 °C	1.0	1.0	2.0
	Charge of DPIP	1.0	2.0	3.0
	Reaction	1.5	3.0	4.5
	Terminate mixing (hold)	1.0	4.5	5.5
	Cooling	10.0	5.5	15.5
	Mixing	0.5	15.5	16.0
	Quality control	1.0	16.0	17.0
	Discharge (to M-01)	2.0	17.0	19.0
	Pressure wash	2.0	19.0	21.0
	Discharge (wash mixture, to V-02)	0.5	21.0	21.5
	Cleaning	2.0	21.5	23.5
M-01	Charge from V-01	2.0	17.0	19.0
	Milling			
	Discharge			
	Cleaning	1.0	19.0	20.0
V-02	Inertize	0.5	16.5	17.0
	Charge from M-01 (& mixing)	2.0	17.0	19.0
	Hold	2.0	19.0	21.0
	Charge from V-01	0.5	21.0	21.5
	Heating to 380 °C	3.0	21.5	24.5
	Reaction & Distillation	2.0	24.5	26.5
	Cooling	6.0	26.5	32.5
	Quality control	1.0	32.5	33.5
	Charge DMAC	2.0	33.5	35.5
	Mixing & Dissolving	1.5	35.5	37.0
	First discharge	1.0	37.0	38.0
	Hold	0.5	38.0	38.5
	Second discharge	1.0	38.5	39.5
	Cleaning	2.0	39.5	41.5

UNIT	OPERATION	OT [h]	Start t [h]	End t [h]
F-01	First charge from V-02	1.0	37.0	38.0
	Filter			
	First discharge			
	Conditioning	0.5	38.0	38.5
	Second charge from V-02	1.0	38.5	39.5
	Filter			
	Second discharge			
	Conditioning	0.5	39.5	40.0
V-03	First charge	1.0	37.0	38.0
	Hold	0.5	38.0	38.5
	Second charge	1.0	38.5	39.5
	Evaporation & Precipitation	3.0	39.5	42.5
	Quality control	2.0	42.5	44.5
	First discharge	0.5	44.5	45.0
	Hold	2.5	45.0	47.5
	Second discharge	0.5	47.5	48.0
	Cleaning	1.0	48.0	49.0
DR-01	First charge from E-01	0.5	44.5	45.0
	Drying	2.0	45.0	47.0
	First discharge	0.5	47.0	47.5
	Second charge from E-01	0.5	47.5	48.0
	Drying	2.0	48.0	50.0
	Second discharge	0.5	50.0	50.5
	Cleaning	0.5	50.5	51.0
H-01	First charge from DR-01	0.5	47.0	47.5
	Storage	2.5	47.5	50.0
	Second charge from DR-01	0.5	50.0	50.5
	Quality control	3.0	50.5	53.5
	Discharge	3.0	53.5	56.5
	Cleaning	0.5	56.5	57.0

UNIT	OPERATION	OT [h]	Start t [h]	End t [h]
M-02	Charge from H-01	3.0	53.5	56.5
	Milling			
	Discharge			
	Cleaning	0.5	56.5	57.0
SP-01	Charge from M-02	3.0	53.5	56.5
	Sieving			
	Discharge to hopper			
	Quality control	1.0	56.5	57.5
	Packaging	5.0	57.5	62.5
	Cleaning	0.5	62.5	63.0

Therefore, the batch time (BT) is extracted from Table 6. Cycle time (CT) is obtained from Table 7. The batch time is the time required to complete a batch, from the beginning to the end of the whole process. On the other hand, the cycle time indicates the time where the overlapping of the first and the second batches is produced and is defined by the bottleneck, V-02. It is important to state that although the cycle time is given by the vessel V-02, operation time of vessel V-01, 23.5 h, is very close to it, and must therefore be kept in mind.

Table 7. Time study

	START t [h]	END t [h]	OT [h]	% of BT
V-01	0	23.5	23.5	37
M-01	17.0	20.0	3.0	5
V-02	16.5	41.5	25.0	40
F-01	37.0	40.0	3.0	5
V-03	37.0	49.0	12.0	19
DR-01	44.5	51.0	6.5	10
H-01	47.0	57.0	10.0	16
M-02	53.5	57.0	3.5	6
SP-01	53.5	63.0	9.5	15

7.2. SCHEDULING

After thoroughly examining the timing of the process, its scheduling must be properly carried out to enhance the plant's efficiency. The plant designed in this study will be shut down for a month in summer, another in wintertime, and three more on other yearly festivities, so, in total, the plant will not be operative for 11 weeks a year. That means it will operate 41 weeks a year, 7 days a week, and 24 hours a day. Therefore, the maximum working time is 6900 hours per year.

As was established in the beginning of this report, the production of the plant is 1000 tonnes/year, and with a batch size of 5 tonnes/batch. Thus, the annual production of the plant is 200 batches per year.

Therefore, the batch time (BT) is extracted from the information given on the previous section, as well as the cycle time (CT). The batch time is the time required to complete a batch, from the beginning to the end of the whole process. On the other hand, the cycle time indicates the time where the overlapping of the first and the second batches is produced.

There are two extreme cases of possible production scheduling of the plant. The first one is to carry out the production in a non-overlapping manner, meaning that a new batch is not started until the previous one has finished. The second one is producing in an overlapping campaign, where the start of the new batch is determined by the equipment considered the bottleneck (cycle time), therefore beginning the new batch before the previous one has ended.

As can be deduced, the non-overlapping option is not very time efficient, while the overlapping alternative is time efficient but leaves no time for solving problems that may arise. Thus, it is convenient to establish a balance between the two and find an adequate middle point, where the overlapping method is chosen but organised in various campaigns.

It can also be observed that in the case of the non-overlapping configuration (Figure 18) the cycle time is the same as the batch time, which in this case, as can be drawn from Table 6, is 63 h.

However, for the overlapping alternative, the batch time, 63 h, differs from the cycle time, which, as shown in Table 7 is 25 h. This situation is represented in Figure 19.

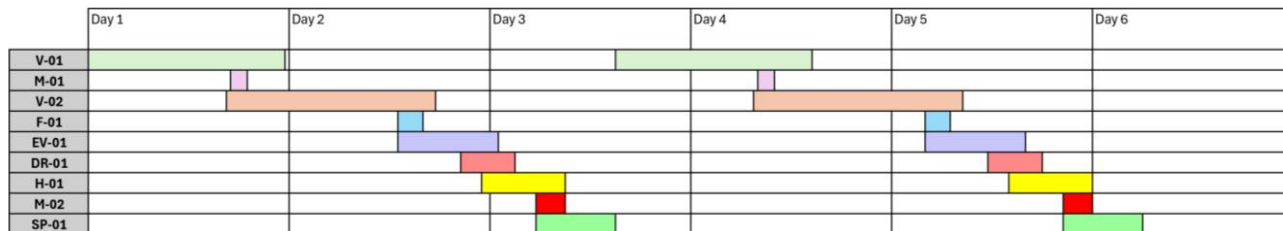


Figure 19. Gantt diagram, non-overlapping configuration

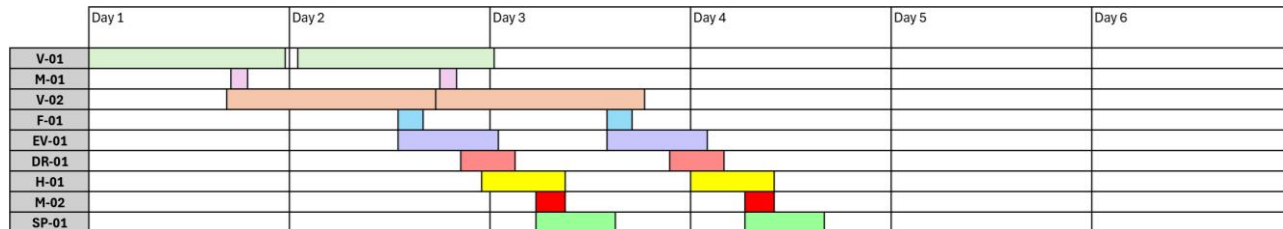


Figure 18. Gantt diagram, overlapping configuration

7.3. PRODUCTION CAMPAIGNS

As mentioned in the previous section, a compromise must be reached between overlapping and non-overlapping configuration. This can be done by choosing the overlapping setting and organising the production in different campaigns. The most adequate number of campaigns can be selected by analysing the different options and comparing them to the available working hours given by the plant calendar.

Therefore, Equation (1) can be used to calculate the makespan of each option, described as the minimum time required to produce a determined number of batches per year. Batch Time (B_T) and Cycle Time (C_T) are those previously set.

$$MT_N = B_T + (N - 1) \cdot C_T \quad (1)$$

Where MT_N is Makespan (h), B_T is the Batch Time (h), N is the batches produced in each campaign, C_T is the Cycle Time (h).

Table 8. Campaign configuration

Campaigns	Batch/Campaign	MT [h]
1	200	5038
2	100	5076
4	50	5152
5	40	5190
8	25	5304
10	20	5380
20	10	5760
25	8	5950
40	5	6520
50	4	6900
100	2	8800
200	1	12600

There are various options of number of campaigns that can be considered, all shown in Table 8. This different examples are presented assuming each batch has the same number of batches for the sake of simplifying the analysis. Nevertheless, that would not necessarily apply in reality, since campaigns could be modified in dependence of market demand distribution through the year.

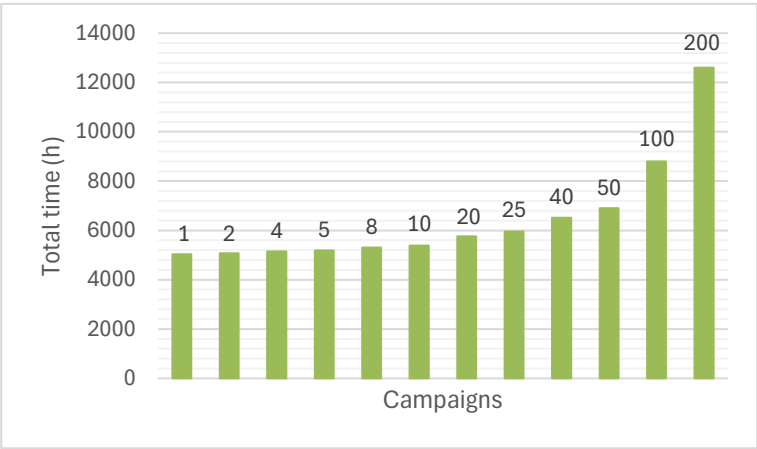


Figure 20. Campaign alternatives duration

As can be observed, the first case is the most time efficient, nevertheless, it raises problems such as the impossibility of machinery maintenance work, both preventive and necessary, increasing the risk of forced shutdown of the process due to equipment malfunction. It also poses a stock issue, since implementing only one campaign denies the possibility of adapting annual production tendencies to market demand, therefore potentially creating a stock that is disengaged with actual client needs.

Nonetheless, the other extreme, of 200 campaigns, is not only impractical, but impossible, since its total time exceeds the theoretical available yearly hours, as well as the cases for 100 and 50 campaigns.

Consequently, the intermediate arrangement of 20 campaigns, and 10 batches/campaign is adopted, which, although it increases the total makespan in comparison to the shortest option, it leaves 9 weeks where the production of PBI will stop, permitting the use of the plant for

another process, as well as a more efficient stocking and the necessary maintenance operations.

7.4. KPIS DETERMINATION

As the final step of the process design, the efficiency of the plant must be analyzed, and to do so, the measures of efficiency (KPIs), must be determined.

Starting by the maximum batches that can be produced per year, considering PBI was the only product for which the production line was destined, and that only one campaign of 5000 tonnes/batch was produced, without stopping the line throughout the year, in the 6900 available hours. This, in turn, permits establishing the maximum yearly production capacity. The minimum production time is the other KPI that must be included, which reflects the time required to produce the annual production of 100 tonnes/year efficiently.

Table 9. KPIs calculation

KPIs	Units	Value
OVERLAPPING		
Maximum number of batches	batch/year	274
Maximum production capacity	tonnes/year	1370
Minimum production time	h/year	5038

8.CONCLUSIONS

From the market research, an estimate of 100 000 tonnes/year is drawn for global PBI production. Based on this, the selected production for the designed plant represents 1 % of the global one, being 1000 tonnes/year.

From the patent study, CA2612722A1 is selected as the main guide. It describes a two-step polycondensation of polybenzimidazole, where the first step, produces an oligomeric precursor of PBI in the form of a friable foam, which is finely pulverized before proceeding to the second step, where the reaction advances until producing a high molecular weight polymer.

The battery limits include synthesis, conditioning and packaging of the product, and exclude raw materials storage, plant services and waste treatment.

The block diagram, mass balance and recipe have been performed considering the process works discontinuously and batch size is 5000 kg.

Calculations and research has been done to design and select the adequate equipment for each step of the designed process. A Process and Instrumentation Diagram (PI&D) has been created.

A thorough time study has been conducted, describing occupation time of each unit in detail. From there, batch time (BT) and cycle time (CT) have been set. A Gantt diagram has been prepared, and different campaign configurations have been studied.

An overlapping disposition of batches, and a total of available time of 6900 h/year, and a work organization of 3 shifts, 24 h/day, and 7 days/week are defined. With these considerations, the conclusion is drawn that the most adequate option for

production are 20 campaigns of 10 batches each, with a total of 200 batches. With this decision, yearly production time amounts to 5760.

Lastly, KPIs have been determined the efficiency of the plant; maximum number of batches, maximum production capacity, and minimum production time.

REFERENCES AND NOTES

1. *The Age of Plastic: From Parkesine to pollution* | Science Museum. Science Museum. <https://www.sciencemuseum.org.uk/objects-and-stories/chemistry/age-plastic-parkesine-pollution#:~:text=Belgian%20chemist%20and%20clever%20marketeer,fully%20synthetic%20plastic%20in%201907> [Accessed March 2024]
2. *Global plastic production* | Statista. (2024). Statista. <https://www.statista.com/statistics/282732/global-production-of-plastics-since-1950/#:~:text=The%20production%20of%20plastic%20requires,molding%20or%20shaping%20the%20plastic> [Accessed March 2024]
3. Das, A., Im, K. S., Kabir, M. M., Shon, H. K., & Nam, S. Y. (2023). Polybenzimidazole (PBI)-based membranes for fuel cell, water electrolysis and desalination. *Desalination*, 579.
4. Powers, E. J., & Serad, G. A. (1986). History and Development of Polybenzimidazoles. En Springer eBooks (pp. 355-373). https://doi.org/10.1007/978-94-011-7073-4_34 [Accessed March 2024]
5. Fishel, K. J., Gullledge, A. L., Pingitore, A. T., Hoffman, J. P., Steckle, W. P., & Benicewicz, B. C. (2016). Solution Polymerization of Polybenzimidazole. *Journal Of Polymer Science*, 54.
6. PubChem. (s. f.-a). 2,2'-m-Phenylene-5,5'-bibenzimidazole. PubChem. <https://pubchem.ncbi.nlm.nih.gov/compound/22058899> [Accessed April 2024]
7. Sannigrahi, A., Ghosh, S., Maity, S., Jana, T. (2010). Polybenzimidazole gel membrane for the use in fuel cell. *Polymer*, 51, 5929–5941.
8. Li, Q., Aili, D., Hjuler, H. A., & Jensen, J. O. (2015). *High Temperature Polymer Electrolyte Membrane Fuel Cells*. Springer.
9. Hearle, J. W. S. (2001). *High-Performance Fibres*. Woodhead Publishing Limited, Cambridge.
10. NASA SpinOff. (2007). *Polymer Fabric Protects Firefighters, Military, and Civilians*.
11. Dawkins, B. G., Baker, J. D., Joiner, R. H., Hudson, K. M. (2006). *A process for a two stage melt polymerization for the production of polybenzimidazole* (Patent No. CA2612722). Canadian Intellectual Property Office.
12. Choe, E. W., & Conciatory, A. B. (1982). *Production of improved high molecular weight polybenzimidazole with tin containing catalyst* (Patent No. 4,452,971). United States Patent Office.
13. Clark, B. K., & Maxwell, R. I. (1958). *Polybenzimidazoles* (Patent No. US2895948A). United States Patent Office.
14. Critchley, J. P. (2003). Polymers, Thermally Stable. *Encyclopedia of Physical Science and Technology* (Third Edition, 775-807).
15. Dahe, G., Singh, R. P., Dudeck, K. W., Yang, D., & Berchtold, K. A. (2019). Influence of non-solvent chemistry on polybenzimidazole hollow fiber membrane preparation. *Journal Of Membrane Science*, 577, 91-103.
16. *High performance Thermoplastic Polymers* | Celazole® PBI Polymer. Celazole® PBI. <https://pbipolymer.com/celazole-pbi-products/> [Accessed April 2024]
17. Sandor, R. B. W. (1990). PBI (Polybenzimidazole): Synthesis, Properties and Applications. *High Performance Polymers*, 2(1), 25-37.

18. Markets - Celazole® PBI. Celazole® PBI. <https://pbipolymer.com/markets/> [Accessed April 2024]
19. Industry Growth Insights. Polybenzimidazole Fiber Market Report | Global forecast to 2028. <https://industrygrowthinsights.com/report/polybenzimidazole-fiber-market/> [Accessed April 2024]
20. Market.us. High Performance Fiber Market Size | CAGR of 8.7%. <https://market.us/report/high-performance-fiber-market/> [Accessed April 2024]
21. Cas 25928-81-8, POLYBENZIMIDAZOLE | lookchem. <https://www.lookchem.com/casno25928-81-8.html> [Accessed May 2024]
22. Polybenzimidazoles (PBI) Market Size, Share, Trends & Forecast. Verified Market Research. <https://www.verifiedmarketresearch.com/product/polybenzimidazoles-pbi-market/> [Accessed May 2024]
23. DataM Intelligence. Polybenzimidazole (PBI) Market Size, Share, Growth, Forecast and Outlook (2024-2031). DataM Intelligence. <https://www.datamintelligence.com/research-report/polybenzimidazole-market> [Accessed May 2024]
24. PubChem. 3,3'-Diaminobenzidine. PubChem. <https://pubchem.ncbi.nlm.nih.gov/compound/7071> [Accessed May 2024]
25. PubChem. Diphenyl isophthalate. PubChem. <https://pubchem.ncbi.nlm.nih.gov/compound/69779> [Accessed May 2024]
26. Diphenyl isophthalate | C20H14O4 | ChemSpider. <https://www.chemspider.com/Chemical-Structure.62978.html> [Accessed May 2024]
27. File:Vapour pressure dimethylacetamide.svg - Wikimedia Commons. https://commons.wikimedia.org/wiki/File:Vapour_pressure_dimethylacetamide.svg [Accessed June 2024]
28. Soldocavitators. Datasheet - soldocavitators. Soldocavitators. <https://soldocavitators.com/datasheet/#tabella> [Accessed June 2024]
29. Molino húmedo. [Video]. Soldo Cavitators - Horizontal / Para Residuos Orgánicos / Para Polvo Grueso. <https://www.directindustry.es/prod/soldo-cavitators/product-236711-2388840.html> [Accessed June 2024]
30. Peiro, S.A. Filtro de bolsa FLOWLINE la solución de filtración más versátil | Peiro, S.A. <https://peiro.com/productos/filtracion/filtros-bolsa/flowline/> [Accessed June 2024]
31. Rota-Cone Vacuum Dryer | Paul O Abbe. <https://www.pauloabbe.com/rota-cone-vacuum-dryer> [Accessed June 2024]
32. Woods, D. R. (2007). *Rules of Thumb in Engineering Practice*. John Wiley & Sons.
33. Ceramic ball mill-Baichy Machinery. [https://www.baichy-com.translate.goog/Powder/ceramic ball mill.html?_x_tr_sl=en&_x_tr_tl=es&_x_tr_hl=es&_x_tr_pto=sc](https://www.baichy-com.translate.goog/Powder/ceramic+ball+mill.html?_x_tr_sl=en&_x_tr_tl=es&_x_tr_hl=es&_x_tr_pto=sc) [Accessed June 2024]
34. Rotary vibrating screen for material screening and filtration. Eversun. <https://www.eversunequipment.com/product/rotary-vibrating-screen/> [Accessed June 2024]
35. FlowMatic®07 big bag filling system. <https://www.palamaticprocess.com/bulk-handling-equipment/big-bag-filling/flowmatic-07> [Accessed June 2024]
36. Green, D. W., & Maloney, J. O. (1997). *Perry's Chemical Engineers' handbook*. McGraw-Hill Professional Publishing.
37. Towler, G., & Sinnott, R. (2012). *Chemical Engineering Design: Principles, Practice and Economics of Plant and Process Design*. Elsevier

ACRONYMS

PBI:	Polybenzimidazole
TAB:	3,3',4,4'-tetraminobiphenyl
DPIP:	Diphenyl isophthalate
V-01:	Reactor vessel 1
M-01:	Humid mill
V-02:	Reactor vessel 2
F-01:	Bag filter
V-03:	Evaporator vessel
DR-01:	Vacuum dryer
H-01:	Hopper 1
M-02:	Ball mill
SP-01:	Sieve and Packaging unit
P&ID:	Piping and Instrumentation Diagram
BT:	Batch Time
CT:	Cycle Time
AFML:	Air Force Materials Laboratory
NASA:	National Aeronautics and Space Administration
KPIs:	Key Performance Indicators
Mw:	Molecular weight
DMAC:	Dimethylacetamide
ZW:	Zero wait
FIS:	Finite intermediate storage

OT: Occupation time

MT: Makespan

APPENDICES

APPENDIX 1: EQUIPMENT DATASHEET

Table 10. Vessel Datasheet V-01

PROJECT: BASIC DESIGN OF PBI PRODUCTION PLANT		Nº:	01
VESSEL			
1	ITEM	V-01	
2	NUMBER OF EQUIPMENT REQUIRED	1	
3	TYPE/POSITION	VERTICAL	
4	SERVICE	PREPOLYMERIZATION	
5	FLUID/S CONTAINED		
6	FASE	-	L/S
7	CORROSIVE and/or TOXIC COMPONENTS	-	T
8	LIQUID DENSITY @P,T	kg/m³	1200
9	OPERATING/DESIGN CONDITIONS		
10	OPERATING TEMPERATURE	°C	25/300
11	OPERATING PRESSURE	bar	ATM
12	DESIGN TEMPERATURE	°C	350
13	DESIGN PRESSURE	bar	1.5
14	CHARACTERISTICS OF THE EQUIPMENT		
15	TYPE	REACTOR VESSEL	
16	MATERIAL	-	AISI 316L
17	DIAMETER	m	2.0
18	HEIGHT	m	4.9
19	VOLUME	m³	15.0
20	THICKNESS	m	0.009
21	AGITATION SYSTEM		
22	TYPE	-	ANCHOR/HELICAL RIBBON
23	HEIGHT	m	2.0
24	STIRRER HEIGHT	m	0.7
25	STIRRER DIAMETER (ANCHOR)	m	1.8
26	STIRRER DIAMETER (HELICAL RIBBON)	m	1.6
27	STIRRER WIDTH	m	0.3
28	JACKET		
29	CONTACT AREA	m2	33.6
30	CONNECTIONS		
31	AGITATOR	N1	

32	NITROGEN	N2
33	PT/PIC	N3
34	RELIEVE VALVE/VENT	N4
35	LSH/LAH	N5
36	LSL/LAL	N6
37	TT/TI	N7
38	OUTLET	N8
39	TT/TIC	N9
40	THERMAL OIL OUTLET	N10
41	THERMAL OIL INLET	N11
42	LIT/LI	N12
43	MANHOLE	N13
44	3,3',4,4'-TETRAMINOBIPHENYL (TAB)	N14
46	DIPHENYL ISOPHTHALATE (DPIP)	N15
46	DEIONIZED WATER	N16

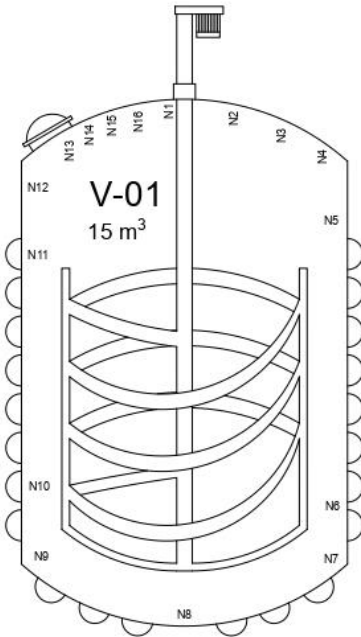


Table 11. Humid Mill Datasheet M-01

PROJECT: BASIC DESIGN OF PBI PRODUCTION PLANT	N°:	02
HUMID MILL		

1	ITEM	M-01	
2	NUMBER OF EQUIPMENT REQUIRED	1	
3	TYPE/POSITION	HORIZONTAL	
4	SERVICE	MILLING	
5	FLUID/S CONTAINED		
6	FASE	-	L/S
7	CORROSIVE and/or TOXIC COMPONENTS	-	-
8	LIQUID DENSITY @P,T	kg/m3	1000
9	OPERATING/DESIGN CONDITIONS		
10	OPERATING TEMPERATURE	°C	25
11	OPERATING PRESSURE	bar	1.5
12	DESIGN TEMPERATURE	°C	200
13	DESIGN PRESSURE	bar	-
14	CHARACTERISTICS OF THE EQUIPMENT		
15	TYPE	HUMID MILL	
16	MATERIAL	-	AISI 316L
17	PARTICLE SIZE OUTPUT	µm	1
18	LENGTH (x)	mm	1000
19	HEIGHT (y)	mm	390
20	DIAMETER (z)	mm	450
21	POWER	kW	4

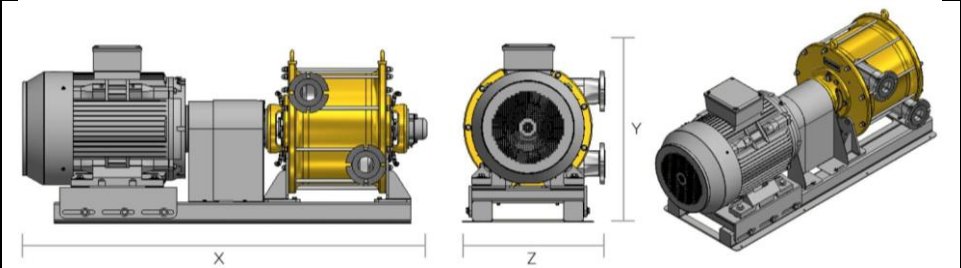


Table 12. Vessel Datasheet V-02

PROJECT: BASIC DESIGN OF PBI PRODUCTION PLANT		Nº:	03
VESSEL			
1	ITEM	V-02	
2	NUMBER OF EQUIPMENT REQUIRED	1	
3	TYPE/POSITION	VERTICAL	
4	SERVICE	POLYMERIZATION	
5	FLUID/S CONTAINED		
6	FASE	-	S/L/G
7	CORROSIVE and/or TOXIC COMPONENTS	-	-
8	LIQUID DENSITY @P,T	kg/m3	1000
9	OPERATING/DESIGN CONDITIONS		
10	OPERATING TEMPERATURE	°C	25/380
11	OPERATING PRESSURE	bar	ATM
12	DESIGN TEMPERATURE	°C	450
13	DESIGN PRESSURE	bar	1.5
14	CHARACTERISTICS OF THE EQUIPMENT		
15	TYPE	REACTOR VESSEL & FLASH DISTILLER	
16	MATERIAL	-	AISI 316L
17	DIAMETER	m	2.4
18	HEIGHT	m	5.8
19	VOLUME	m3	25.0
20	THICKNESS	m	0.009
21	AGITATION SYSTEM		
22	TYPE	-	ANCHOR/HELICAL RIBBON
23	HEIGHT	m	2.4
24	STIRRER HEIGHT	m	0.8
25	STIRRER DIAMETER (ANCHOR)	m	2.2
26	STIRRER DIAMETER (HELICAL RIBBON)	m	1.9
27	STIRRER WIDTH	m	0.3
28	JACKET		
29	CONTACT AREA	m2	46,7
30	CONNECTIONS		
31	AGITATOR	N1	
32	NITROGEN	N2	
33	PT/PIC	N3	
34	RELIEVE VALVE/VENT	N4	

35	DISTILLATON (TO CONDENSER)	N5
36	LSH/LAH	N6
37	LSL/LAL	N7
38	TT/TI	N8
39	OUTLET	N9
40	TT/TIC	N10
41	THERMAL OIL OUTLET	N11
42	THERMAL OIL INLET	N12
43	LIT/LI	N13
44	MANHOLE	N14
45	PREPOLYMER WASH	N15
46	HUMID PREPOLYMER	N16
47	DMAC	N17
48	DEIONIZED WATER	

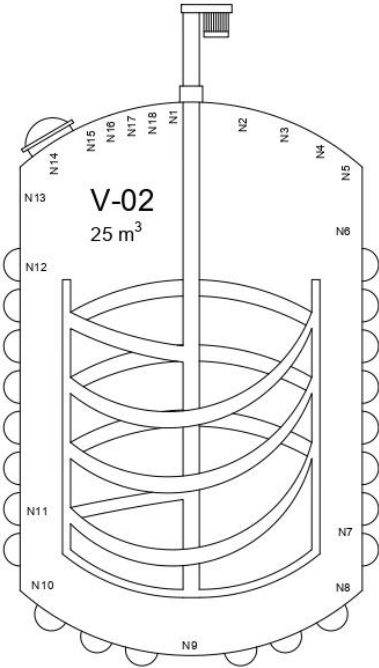


Table 13. Filter Datasheet F-01

PROJECT: BASIC DESIGN OF PBI PRODUCTION PLANT		Nº:	04
BAG FILTER			
1	ITEM	F-01	
2	NUMBER OF EQUIPMENT REQUIRED	1	
3	TYPE/POSITION	VERTICAL	
4	SERVICE	FILTRATION	
5	FLUID/S CONTAINED		
6	FASE	-	L/S
7	CORROSIVE and/or TOXIC COMPONENTS	-	-
8	LIQUID DENSITY @P,T	kg/m3	940
9	OPERATING/DESIGN CONDITIONS		
10	OPERATING TEMPERATURE	°C	100
11	OPERATING PRESSURE	bar	ATM
12	DESIGN TEMPERATURE	°C	120
13	DESIGN PRESSURE	bar	10
14	CHARACTERISTICS OF THE EQUIPMENT		
15	TYPE	BAG FILTER	
16	MATERIAL	-	AISI 316L
17	BODY HEIGHT (C)	mm	1051
18	LEG HEIGHT (F)	mm	485
19	BAG DIAMETER	mm	180
20	BAG HEIGHT	mm	810

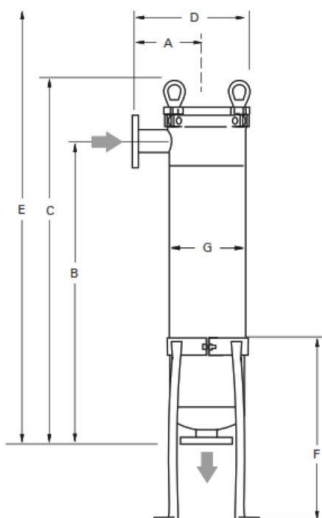


Table 14. Vessel Datasheet V-03

PROJECT: BASIC DESIGN OF PBI PRODUCTION PLANT		Nº:	05
VESSEL			
1	ITEM	V-03	
2	NUMBER OF EQUIPMENT REQUIRED	1	
3	TYPE/POSITION	VERTICAL	
4	SERVICE	EVAPORATION & PRECIPITATION	
5	FLUID/S CONTAINED		
6	FASE	-	L/S
7	CORROSIVE and/or TOXIC COMPONENTS	-	-
8	LIQUID DENSITY @P,T	kg/m3	940
9	OPERATING/DESIGN CONDITIONS		
10	OPERATING TEMPERATURE	°C	180
11	OPERATING PRESSURE	bar	ATM
12	DESIGN TEMPERATURE	°C	200
13	DESIGN PRESSURE	bar	1.5
14	CHARACTERISTICS OF THE EQUIPMENT		
15	TYPE	EVAPORATOR VESSEL	
16	MATERIAL	-	AISI 316L
17	DIAMETER	m	2.4
18	HEIGHT	m	5.8
19	VOLUME	m3	25.0
20	THICKNESS	m	0.009
21	AGITATION SYSTEM		
22	TYPE	-	ANCHOR/HELICAL RIBBON
23	HEIGHT	m	2.4
24	STIRRER HEIGHT	m	0.8
25	STIRRER DIAMETER (ANCHOR)	m	2.2
26	STIRRER DIAMETER (HELICAL RIBBON)	m	1.9
27	STIRRER WIDTH	m	0.3
28	JACKET		
29	CONTACT AREA	m2	46,7
30	CONNECTIONS		
31	AGITATOR	N1	
32	NITROGEN	N2	
33	PT/PIC	N3	
34	RELIEVE VALVE/VENT	N4	
35	DISTILLATON (TO CONDENSER)	N5	
36	LSH/LAH	N6	
37	LSL/LAL	N7	

38	TT/TI	N8
39	OUTLET	N9
40	TT/TIC	N10
41	THERMAL OIL OUTLET	N11
42	THERMAL OIL INLET	N12
43	LIT/LI	N13
44	MANHOLE	N14
45	DMAC & PBI SOLUTION	N15
46	DEIONIZED WATER	N16
47	-	N17

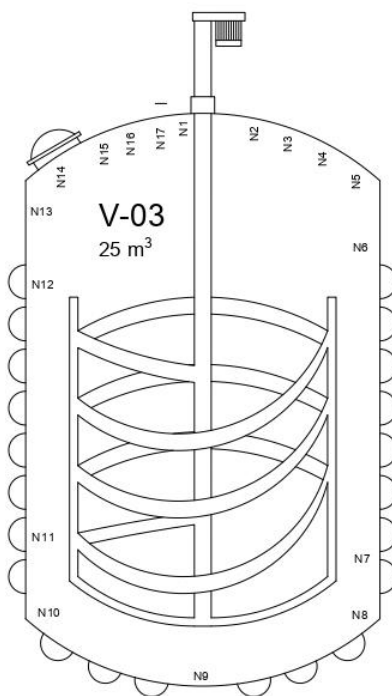


Table 15. Vacuum Dryer Datasheet DR-01

PROJECT: BASIC DESIGN OF PBI PRODUCTION PLANT		Nº:	06
VACUUM DRYER			
1	ITEM	DR-01	
2	NUMBER OF EQUIPMENT REQUIRED	1	
3	TYPE/POSITION	VERTICAL	
4	SERVICE	VACUUM DRYING	
5	FLUID/S CONTAINED		
6	FASE	-	S/L
7	CORROSIVE and/or TOXIC COMPONENTS	-	-
8	LIQUID DENSITY @P,T	kg/m3	940
9	OPERATING/DESIGN CONDITIONS		
10	OPERATING TEMPERATURE	°C	60
11	OPERATING PRESSURE	bar	ATM/0.01
12	DESIGN TEMPERATURE	°C	200
13	DESIGN PRESSURE	bar	0.001/1.1
14	CHARACTERISTICS OF THE EQUIPMENT		
15	TYPE	VACUUM DRYER	
16	MATERIAL	-	AISI 316L
17	DIAMETER (A)	m	2.30
18	OVERALL HEIGHT (H)	m	3.58
19	JACKETED SURFACE	m2	16.17
20	ROTATION SPEED	rpm	3
21	WORKING CAPACITY	m3	3.90

The technical drawing illustrates the dimensions of the vacuum dryer. The front view on the left shows a circular cross-section with a central agitator shaft and a conical base. The side view on the right shows the vertical profile of the cylindrical vessel. Dimension labels are as follows: A is the internal diameter; B is the internal height; C is the top flange thickness; D is the bottom flange thickness; E is the total height; F is the height to the top of the vessel; G is the height to the bottom of the vessel; H is the overall height including the base; I is the diameter of the top flange; J is the width of the base; K is the width of the base; L is the length of the base; M is the total length including the base; N is the height of the base.

Table 16. Ball Mill Datasheet M-02

PROJECT: BASIC DESIGN OF PBI PRODUCTION PLANT		Nº:	07
BALL MILL			
1	ITEM	M-02	
2	NUMBER OF EQUIPMENT REQUIRED	1	
3	TYPE/POSITION	HORIZONTAL	
4	SERVICE	MILLING	
5	FLUID/S CONTAINED		
6	FASE	-	S
7	CORROSIVE and/or TOXIC COMPONENTS	-	-
8	LIQUID DENSITY @P,T	kg/m3	-
9	OPERATING/DESIGN CONDITIONS		
10	OPERATING TEMPERATURE	°C	40
11	OPERATING PRESSURE	bar	ATM
12	DESIGN TEMPERATURE	°C	200
13	DESIGN PRESSURE	bar	-
14	CHARACTERISTICS OF THE EQUIPMENT		
15	TYPE	BALL MILL	
16	MATERIAL	-	AISI 316L
17	PARTICLE SIZE OUTPUT	µm	100-200
18	LENGTH	mm	1800
19	DIAMETER	mm	900
20	POWER	kW	18.5



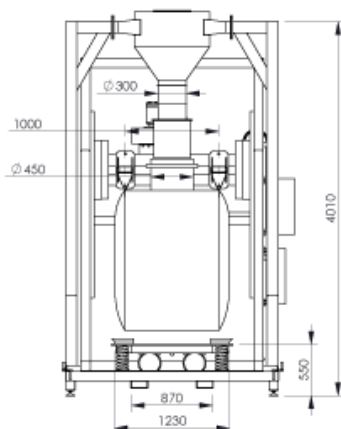
Table 17. Sieve Datasheet SP-01

PROJECT: BASIC DESIGN OF PBI PRODUCTION PLANT		Nº:	08
SIEVE			
1	ITEM	SP-01 (Sieve)	
2	NUMBER OF EQUIPMENT REQUIRED	1	
3	TYPE/POSITION	VERTICAL	
4	SERVICE	SIEVING	
5	FLUID/S CONTAINED		
6	FASE	-	S
7	CORROSIVE and/or TOXIC COMPONENTS	-	-
8	LIQUID DENSITY @P,T	-	-
9	OPERATING/DESIGN CONDITIONS		
10	OPERATING TEMPERATURE	°C	40
11	OPERATING PRESSURE	bar	ATM
12	DESIGN TEMPERATURE	°C	-
13	DESIGN PRESSURE	bar	-
14	CHARACTERISTICS OF THE EQUIPMENT		
15	TYPE	VIBRATING SIEVE	
16	MATERIAL	-	SUS 316L
17	DIAMETER	m	2.0
18	EFFECTIVE SCREENING AREA	m2	2.9
19	SCREEN MESH	µm	90-200
20	POWER	kW	4.5



Table 18. Packaging Datasheet SP-01

PROJECT: BASIC DESIGN OF PBI PRODUCTION PLANT		Nº:	09
BIG BAG FILLING SYSTEM			
1	ITEM	SP-01 (Packaging)	
2	NUMBER OF EQUIPMENT REQUIRED	1	
3	TYPE/POSITION	VERTICAL	
4	SERVICE	PACKAGING	
5	FLUID/S CONTAINED		
6	FASE	-	S
7	CORROSIVE and/or TOXIC COMPONENTS	-	-
8	LIQUID DENSITY @P,T	-	-
9	OPERATING/DESIGN CONDITIONS		
10	OPERATING TEMPERATURE	°C	25
11	OPERATING PRESSURE	bar	ATM
12	DESIGN TEMPERATURE	°C	-
13	DESIGN PRESSURE	bar	6
14	CHARACTERISTICS OF THE EQUIPMENT		
15	TYPE	BIG BAG PACKAGING	
16	MATERIAL	-	SS 316L
17	FILLER DIAMETER	mm	450
18	HEIGHT	mm	4010
19	WEIGHING PRECISION	g	
20	POWER	kW	1.7



APPENDIX 2: TABLE CONTENT

Table 1. Polybenzimidazole properties	3
Table 2. Patent reasearch	14
Table 3. Raw materials properties [24, 25, 26]	18
Table 4. Equipment losses	22
Table 5. Mass balance	23
Table 6. Ocupation times study	46
Table 7. Time study	48
Table 8. Campaign configuration	53
Table 9. KPIs calculation	55
Table 10. Vessel Datasheet V-01	64
Table 11. Humid Mill Datasheet M-01	65
Table 12. Vessel Datasheet V-02	67
Table 13. Filter Datasheet F-01	69
Table 14. Vessel Datasheet V-03	70
Table 15. Vacuum Dryer Datasheet DR-01	72
Table 16. Ball Mill Datasheet M-02	73
Table 17. Sieve Datasheet SP-01	74
Table 18. Packaging Datasheet SP-01	75

APPENDIX 3: FIGURE CONTENT

Figure 1. Poly[2,2'-(m-phenylene)-5,5'-bibenzimidazole] structure [14].	2
Figure 2. General PBI polycondensation [12]	4
Figure 3. Solvent free PBI synthesis [9]	4
Figure 4. Homogenous solution PBI synthesis [8]	5
Figure 5. Global High Performance Fibers Market Forecast [20]	8
Figure 6. Global PBI Fiber Market Forecast [19]	9
Figure 7. Battery limits	15
Figure 8. Boiling point DMAC vs. Temperature [27].	18
Figure 9. Block diagram plant	19
Figure 10. Anchor and helical ribbon agitator.	32
Figure 11. Humid mill [33]	33
Figure 12. Bag filter [30]	34
Figure 13. Vacuum dryer [31].	36
Figure 14. Hopper configuration	36
Figure 15. Ball mill [33]	37
Figure 16. Sieve [34]	38
Figure 17. Packaging unit [35]	38
Figure 18. Gantt diagram, non-overlapping configuration	51
Figure 19. Gantt diagram, overlapping configuration	51
Figure 20. Campaign alternatives duration	54

