POLITECNICO DI TORINO

Department of Electronics and Telecommunication Engineering

Electronic Engineering (Embedded Systems)

Master's Degree Thesis

Digital temperature measurement system for industrial mixers

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Thank you.

Dario Felice

Summary

Nowadays, the mixing operation is one of the most common processes in the consumer industry for liquid products which involve physical and chemical change. Johnson & Johnson, one of the biggest multinational company active in this sector, uses this process on a large scale to produce its health consumer products - like creams and shampoos. Although much knowledge on mixing has developed from chemical, food and pharmaceutical industries the components of the mixer have been studied to optimize the time to reach homogenization or in order to save power, but not to reach a homogeneous temperature in the blending fluid. Indeed, in these processes the temperature is increased or decreased according to the raw material used to facilitate the blending and melting. In fact, some processes in Johnson & Johnson require up to 85°C. Consequently, this leads to the incorporation of a sensor that controls the heating system according to the temperature of the fluid. The sensor, that is currently used, is neither able to track the entire temperature in the machine nor is it able to save the data for future studies. This means that the sensor can only determine the temperature at its precise point in the machine and not the temperature at any other points. The temperature of the fluid is not homogeneous due to the structure of heating system and the big dimensions of the tank. Some industrial mixers can produce up to 16 tons of products.

Therefore, the purpose of this thesis is to design and develop a low power temperature measurement system that is able to collect the temperature of the fluid in multiple points inside the mixers. The system is developed and produced in the Johnson & Johnson consumer plant located in Pomezia.
A possible wireless sensoring system in the fluid has been studied both to avoid cables inside the mixer and to make it possible to integrate the system into environments that have specific requirements about compatibility with the chemical materials used during the mixing process. Unfortunately, the conductivity of the products does not allow for wireless communication with a range greater than 10 cm between the sensors. For this reason, the entire design integrates both wired and wireless communications and it is studied to respect the Good Manufacturing Procedure (GMP) requirements - including chemical and thermal compatibility.

In order to obtain more flexibility for future modifications, for instance by adding new sensors for ph measurements, and to maintain costs low, the Arduino™ boards with LoRa® wireless communication have been chosen as computational units. These boards are realized for low energy systems that require wireless communication.
As a temperature sensor chemically compatible with the environment where it shall be inserted, sensor DS18B20 configured as waterproof has been purchased. The principle of this kind of sensor is based on forward voltage drop of a silicon diode which is temperature dependent and, furthermore, it is based on 1-wire protocol which has as an advantage a limited number of wires for all sensors. The system measures the temperature only when the mixer works, to save power, this means that the motion sensors are incorporated in the anchor to detect its motion and therefore they can start measuring the temperatures.
Each sensor is tested in laboratories and, through the Minitab tool, the results are collected to observe if they respect the requirements imposed by the company.

After the realization of the system, it is tested in different conditions and in a pilot mixing machine (the small reproduction of the industrial mixer). The initial studies have been performed to observe the temperature gradients according to the speed of the propeller and impeller embedded in the mixer and according to the blending fluid. Indeed, in the results, viscosity and flow are observed as parameters which influence the homogenization of the temperature. The higher the viscosity the higher the temperature differences between the sensors, while turbulent flow homogenizes the temperature faster than laminar flow. Indeed, with the same speed of anchor (2,5 rpm) the time taken for the water to heat is shorter than the time taken to heat the fluid with viscosity around 3000 cps. Indeed, as shown in figure 1, after the same space of time the temperature in water is rather homogeneous and it is around 32°C, while in the viscosity product the average temperature is around 42°C with a maximum temperature gradient of around 20°C.

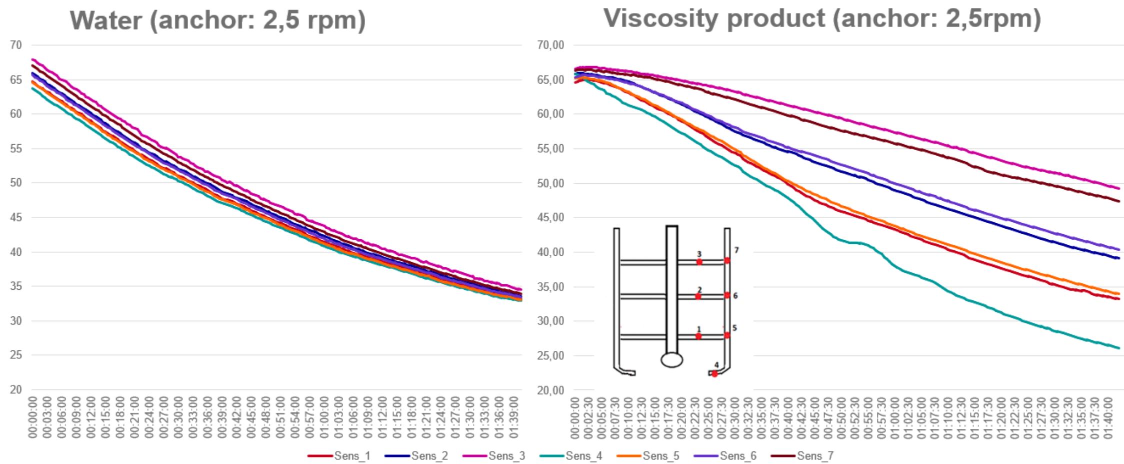

Figure 1. Comparison between water and viscosity product

In tests involving water the effects of flow are observed, in fact in laminar flow the temperature gradients can be observed while in turbulent flow the temperature inside the mixer is homogeneous.

The system is able to detect the temperature gradients of the fluid, viscous or not, inside the mixer and it is able to report the data collected in a computer using excel tool. Therefore the prototype respects the design requirements and it can be used for future analysis regarding fluid dynamics simulations to optimize the process and the hardware of the mixing.

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Chapter 1

Introduction

Johnson & Johnson Consumer is an American multinational company active in the consumer health sector including baby care, oral health and skin & hair care. Many of these products are the results of the blend between different materials, meaning that they are produced with a mixing process.
This thesis describes the development of a temperature measurement system, starting from the design going onto its realization and finally onto the tests phase. The system will be able to measure the temperature at different points inside an industrial mixer in order to observe and study the heat transfer during a mixing process to be able to increase the performance of the process in terms of quality and cost. To understand the need of this measurement system and the process behind a health care product based on a blend of materials, a small but thorough explanation about the industrial mixing procedure, from the process to the mechanical parts, is given to the reader in this first chapter. Then the temperature issue which affects the mixing production is described in order to understand the limitations of the system nowadays. Lastly the V-model is explained as the guideline followed for this project according to the policy of the company in which the project was developed.

1.1 Mixing process introduction

Mixing is a unit operation that is widely practiced to meet a variety of process requirements. The specific mixing system design, operating arrangement and power requirements depend largely on the desired form of the intermediate or final products[1]. Thus, mixing is a key process in the pharmaceutical, chemical and related industries, in fact, inadequate mixing process can result in unsatisfactory product quality and increased production cost. It is central to the extent that, at a recent

[1]Nicholas P. Cheremisinoff, Handbook of Chemical Processing Equipment, Elsevier, 2000, pp 435

meeting of industrialists and academics in the USA, it was estimated that the cost to their process industries due to an inadeguate understanding of mixing was of the order of US$ 1 to 10 billion per annum[2].

In this work, the common mixing process is considered, which is based on batch fluid mixing. This is the technology of blending fluid substances, including gases and solids during a batch operation. In this process, all ingredients are loaded into the mixer together or in a pre-defined sequence and mixed until a homogeneous material is produced and discharged from the mixer in a single lot, called batch. The above-mentioned results achievable with this mixing process are different and the most famous are:

- creating a suspension of solid particles;
- the blending miscible or immiscible liquids;
- promoting heat transfer between a liquid and the jacket of a heat exchanging device.

For instance, in producing an emulsion, a blend of immiscible liquids, as explained later, a sufficient energy is employed to "break up" the dispersed phase, so an high shear stresses are developed in the mixing medium. These kinds of stresses depend on velocity gradients in the blending process that reduce concentration and temperature gradients increasing the overall rates of mass and heat transfers.

The most important characteristics of all mixers that provide a basis for comparative evaluations are the following two:

- the efficiency of the device: it characterizes the quality of process to be treated and may be expressed differently depending on the mixing purpose;
- the mixing intensity: it is determined by the time required to achieve a desired technological result.

So, from an economic standpoint, it is beneficial to achieve the required mixing effect in the shortest possible time, which leads to a decrease of the total energy consumption during mixing operation.
Mechanical mixers are employed almost exclusively when blending liquids and they comprise mostly rotating devices.[3]

[2]A.W. Niemow, M.F. Edwards, N. Harnby, Mixing in the process industries, Butterworth-Heinemann, 1997, pp 1

[3]Nicholas P. Cheremisinoff, Handbook of Chemical Processing Equipment, Elsevier, 2000, pp 167

1.2 Mixer: mechanical and production

Mechanical mixer devices are made up of few simple different parts. The first one is the vessel, a vertically mounted cylindrical tank which will be typically filled to a depth equal to about one tank diameter when running full. Its shape is designed to increase the performance of the entire process according to the amount of product required. In Johnson & Johnson plants, the vessels are realized with stainless steel AISI 316L, its primary alloying constituents after iron, are chromium (between 16-18%), nickel (10-12%) and molybdenum (2-3%).

Another fundamental part of the mixer is the impeller that constitutes the working element of the apparatus, integrated on a vertical, horizontal or inclined shaft. The impellers embedded in the considered tester and industrial mixers are two: a propeller resembling a marine one and paddle mixer in anchor configuration, the figure 1.1. shows an example of both. The propeller and the anchor impeller can work together at the same time[4].

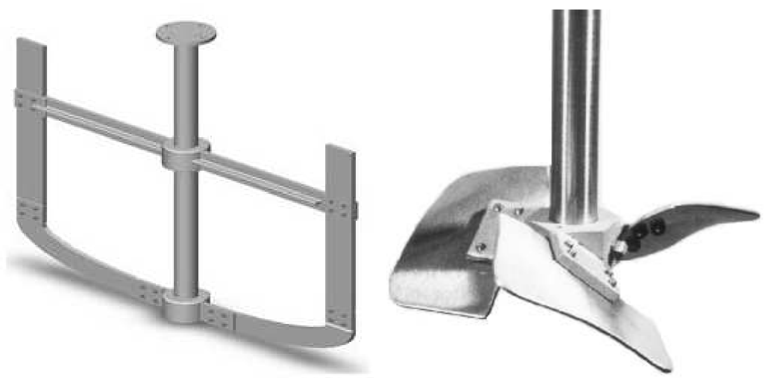

Figure 1.1. Embedded impellers: A anchor impeller, B propeller

The anchor diameter is almost as large as the inside diameter of the tank in such applications, so that the outer and bottom edges of the paddle scrape (or clean) the walls and the bottom. In Johnson & Johnson production process, the anchor paddle has a rotation velocity included into 2-20 rpm creating low convection and circular mixing while the propeller is used to force convection at about 700-900 rpm. The shearing action is so obtained in blending thanks to the high velocity gradient (a liquid has the same velocity of the wall).
During a common mixing process, the raw materials (e.g. distilled water, flavours, oil, etc.) are inserted in the mixer, through external pipes, from two positions:

[4] A. Almasi, the flow of fluids in industrial mixers, April 2, 2019, article published on "Flow Control", www.flowcontrolnetwork.com

either the top or the bottom. The largest quantities of liquids are inserted from the lowest part, using pumps and valves, while some liquids and solid materials are poured form the upper part by hand or by mechanical pumps.

In many blending processes the temperature is used to help the melting of raw materials in water. Increasing the temperature of a big mixer content takes a long time whilst decreasing the temperature often takes even longer. These processes usually have a maximum temperature equal to 85°C. Indeed, while many mixing devices are designed on the basis of blending, mass transfer, and chemical reaction, in some cases heat transfer is the controlling mechanism, especially on the largest scales of operation[5]. Thus, for instance, the rotational speed of an impeller in a mixing vessel may be selected to satisfy a given thermal duty and the agitation conditions so generated may be more than enough for the mixing duty.
In mixers installed in the plant, to increase or decrease the temperature of the internal fluid in the mixer, a circular half-pipe, which has stream or hot/cold water flows inside, is applied all around the vessel. The half-pipe system is shown in figure 1.2. The temperature of hot water, which is at high pressure, is around 135°C while the temperature of cold water is around 15°C. This heat system is controlled by a single temperature sensor positioned internally at the bottom of the vessel.

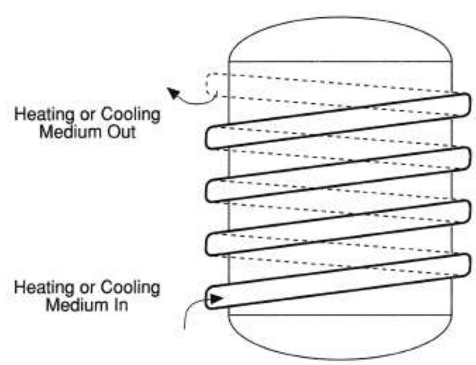

Figure 1.2. Heat transfer with half-pipe approach

The health care products produced by Johnson & Johnson with a mixing process is divided in three main categories:

[5] A.W. Niemow, M.F. Edwards, N. Harnby, Mixing in the process industries, Butterworth-Heinemann, 1997, pp 5

- Cleansers: they refer to a product that cleans or removes dirt or other substances. They are blends of surfactants[6] in a unique liquid solution e.g. shampoo and shower gel. The composition is shown in figure 1.3.

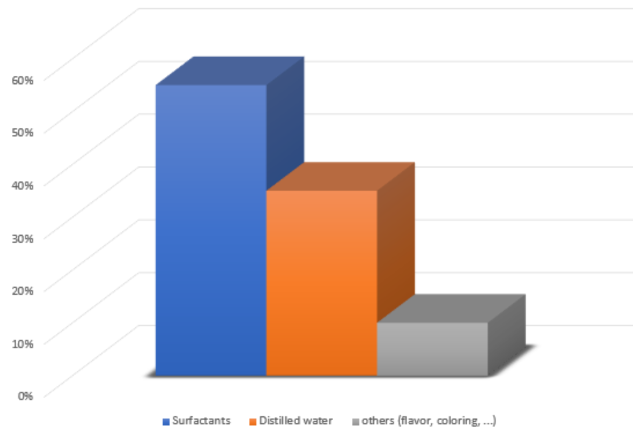

Figure 1.3. Composition of cleanser

- Emulsions: biphasic or multiphasic blends composed of oil and mainly water. They tend to have a cloudy appearance because the many phase interfaces scatter light as it passes through the emulsion. The emulsions are frequently used in pharmaceutics and cosmetics. The emulsion composition is shown in figure 1.4.

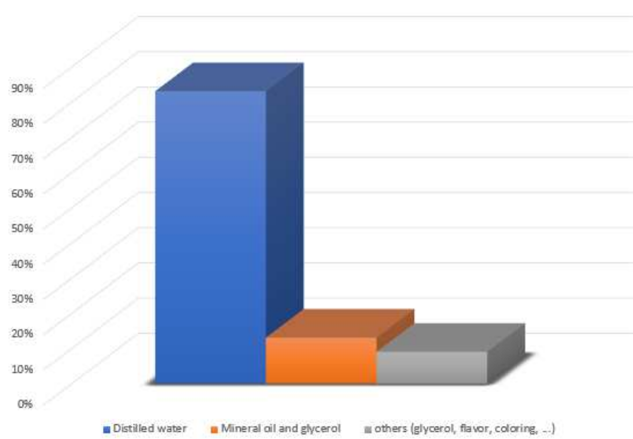

Figure 1.4. Composition of emulsions

[6]Surfactant, also called surface-active agent, substance such as a detergent that, when added to a liquid, reduces its surface tension, thereby increasing its spreading and wetting properties.

- Mouthwashes: blend with high percentage of water and alcohol used for teeth cleaning. Usually they are antiseptic solutions intended to reduce the microbial load in the oral cavity. The composition is shown in figure 1.5.

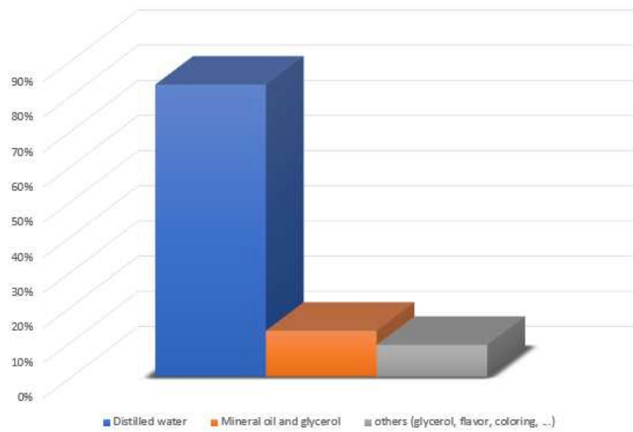

Figure 1.5. Composition of mouthwashes

The first two products are produced using a "hot" process, where, as said before, the temperature is increased to help the melting, while the last one is a "cold" process. Therefore during the production of mouthwashes the temperature of the mixer is neither increased nor decreased, so no temperature control is used, for this reason it is not taken into consideration in this dissertation. For the cleansers, the maximum temperature reached is around 70°C while for the emulsion it is around 85°C.

1.2.1 Mixer: temperature issue

As reported earlier, temperature is a key parameter in the mixing process, it helps the melting of the substance, especially solid raw materials, or it needs to be lower than a target so as not to ruin the raw material inserted. For example, the fragrance cannot be added if the temperature of the fluid is greater than 30°C to avoid a change in its scent.
A time (t) and temperature (T) graph is reported in figure 1.6. for a typical process and a nowadays process to show the temperature variation during the blending operation.

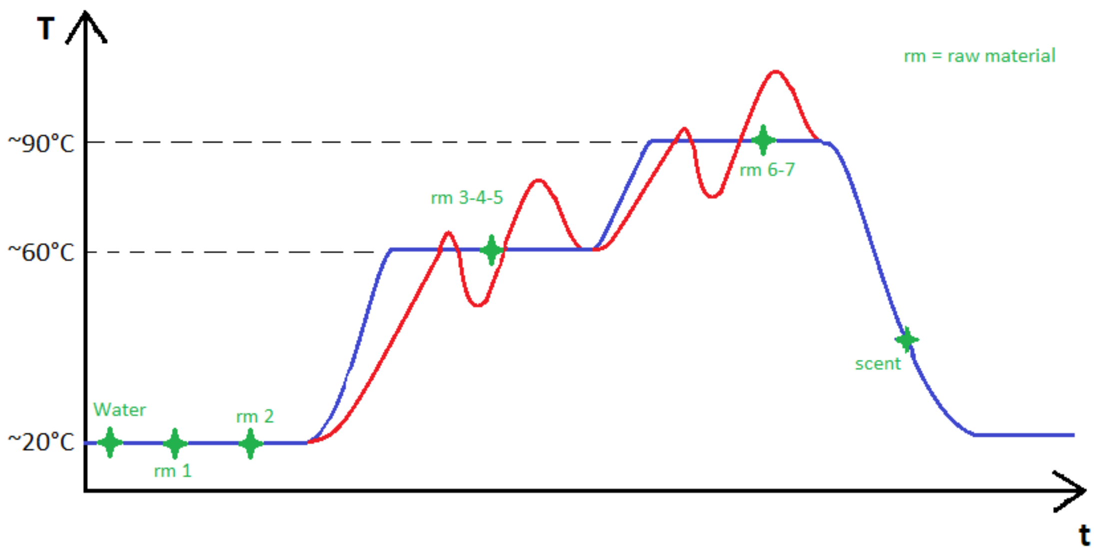

Figure 1.6. Example of process: time-temperature graph

The blue line is the ideal process in which the temperature is constant in the whole fluid during the mixing process and the temperature slope is equal to a straight line. The red part in the graph is taken from the unique control temperature sensor, implemented nowadays, in the bottom mixer. This sensor is a PT100 and it is the only temperature measurement system in the industrial and pilot mixer of the company. The usage of single temperature sensor leads to elongations, indeed if a colder material is added near the sensor, it will read a rapid decrease of temperature opening the valves of the heating system and will bring the average temperature of fluid to being greater than the target point. This can be seen as wasted time and a decrease in performance.
Moreover, the steam in the half-pipe heat device is loaded from the top, this can lead to a higher temperature at the top in compared to the bottom where the steam will have a lower temperature, even if nothing is added. This is only a hypothesis, because the measurement device is not able to scan the occurrence of this effect.
Thus, the single sensor is not able to catch the temperature gradients and the temperature in a single point cannot represent the temperature in the whole fluid. Another issue is the history of data, indeed no measurement is saved; they are only used to control of the heating system and thus no studies were ran to optimize the process from this point of view.

A real-time temperature measurement in different points inside the mixer can overcome these problems. Indeed, the temperature fluid dynamics can be studied to improve the mixing process, by changing the speed of the impeller and turning on/up the heating system, if the average temperature is equal to the set-point, though the bottom sensor measures a lower temperature. This leads to a minor power consumption and less process time avoiding the waste of time and improving the heat fluid transfer. Furthermore, the knowledge of the temperature variation in a mixing cycle can improve the quality of the final products. For example, at the time when the fragrance is added or other materials which require precise temperature to not be damaged. All these aspects lead a big cost saving for a company based on products produced with the batch fluid mixing process.

During the tests of the system, which will reproduce thermal cycle and real production in pilot mixer, the temperature gradients in height and width are expected. The temperature differences, inside the mixer, will depend on various parameter including the viscosity of the fluid and the flow motion.
The heat transfer coefficient depends on the viscosity, therefore between the water and the products, which have the viscosity around $2500 - 3500cps$, the gradients will be different. Higher viscosity means higher temperature difference.
Moreover lower temperature gradients is expected when the flow is turbulent instead of laminar. Therefore in low speed the temperature difference will be very large and with the increasing of speed there will be the decrease of temperature difference.

1.3 Project design: V-model

In this essay, the design of the entire project is arranged following the called "V-model", shown in the figure 1.7, according to the company policy. This model is a unique, linear development methodology used during a software development life cycle and then it is updated and modified by each company so that it can be adapted it for any project. In fact, it is the most common usage in the modern industry companies, thanks to its simplicity and hierarchical shape. The V-model focuses on a typical waterfall method that follows strict, step-by-step stages.

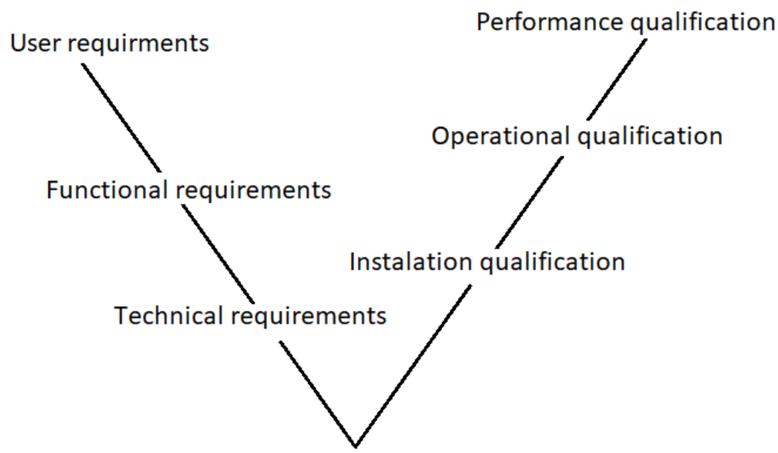

Figure 1.7. Project V-diagram

Following the V-model, on the top-left hand corner, as a first step, we find the user requirements. This section consists of what the system will do, thus the user requirements describe the business needs for what users require from the system. As second step, we find the functional requirements being elaborated, and this part explains how the system works. These requirements defines a function of a system or its component, where function is described as a specification of behavior between outputs and inputs[7]. As a last step of the first arm, the specification stream, the technical details are arranged, so how the functional requirements are met. They are the technical issues that must be considered to successfully complete a project. These are aspects such as performance, reliability and availability that a project must meet on in order to proceed with it.
The bottom angle represents the implementation of the project. It includes hardware and software which respect the requirements listed above.
The right arm includes all tests to qualify and validate the whole system. The testing stream contains the Installation Qualification (IQ) where the physical design is qualified, thus, the connections are tested and the mounted components are verified if they are the required ones. Then there is the Operational Qualification (OQ) in which the components are verified, they need to work correctly as they are designed and programmed. As the last step, Performance Qualification (PQ) is computed. In this step the whole system is tested in general and in detail. After it, the system is ready to work as designed without issues or problems and the project can be signed as being complete.

[7]Fulton R, Vandermolen R (2017). "Chapter 4: Requirements - Writing Requirements". Airborne Electronic Hardware Design Assurance: A Practitioner's Guide to RTCA/DO-254. CRC Press. pp. 89–93. ISBN 9781351831420. Retrieved 15 June 2018

1.3.1 User requirements

The first question about the system is: what will it do? The answer is a simple list of wanted results in general form without precise specifications. In this project the user requirements can be listed as follow:

- Cartesian graph of temperature in function of time up to 9 points inside the mixer;
- The collected data will be available to laboratory technicians and to remote with a computer;
- Data collection in real-time historically;
- No frequent access into the mixer;
- Good Manufacturing Practice (GMP) hardware with small modifications of the mixer;
- Precise and reliable measurements.

These requirements are decided by the people who commission the project. They seem to be very simple but as we can observe in the next subsection, they became strictly functional requirements after the translation.

1.3.2 Functional requirements

The user requirements listed before are translated in the functional requirements, where more details are added to understand how the system will work, without specifying the components or the method used to get the results. These requirements are written with the collaboration of people who commission the project and the people who design and realize the system.
The first user requirements can be translated in the following way: all the data collected from the sensors must be grouped together in a table to simplify reading and also shown on a monitor by means of Cartesian graph where temperatures are plotted with different colours. The first user requirement is also able to give information on the number of sensors which should be maximum 9. In the same way all previous user requirements shall be translated in functional requirements. Those regarding temperature are summarized below:

- Range: the temperature of the liquid in the blending process is in the range between 15°C and 85°C. This is the actual temperature involved in the nowadays mixing process, but to be ensure that the system will be able to work also with a new processes that could involve higher or lower temperatures, the temperature measurement ranges from 15°C to 95°C;

- Resolution: the resolution required is about 1°C to allow a good control and collecting of data for the temperature fluid study realized in the following months after the conclusion of the project. The resolution is decided according to the resolution required for the single temperature sensor mounted in the bottom of the vessel;

- Accuracy: the accuracy required for the actual sensor implemented in the mixer, used as control parameter, is equal to 1°C, so for the project the same accuracy is required;

- Repeatability: it is a measure of a system's consistency to achieve identical results across multiple tests under the same conditions. Its weight in the gage evaluation will be less than to 2% according to the company policy;

- Reproducibility: it is a measure of a system's consistency to achieve identical results across multiple tests under different conditions. Its weight in the gage evaluation will be less than to 2% according to the company policy;

- Sampling frequency: the entire system performs the computation of temperature measurement every 30 seconds ($f = 1/t = 33{,}33 mHz$) that is an optimal trade-off between the sampling for control and the one to maintain low the power consumption. In fact a minor sampling time can be translated in a minor battery life-time, while a greater time can lead issues to the control of the heating system.

The battery life-time will be about one and a half years and it is the critical requirement of the system to avoid frequent access to the mixer which is classified as confined areas[8].
The Good Manufacturing Practice (GMP) imposes the use of accepted material to avoid problem of wear and contamination of the product. This also means that the system will have to respect the cleaning requirements so that it is automatically washed completely during the automatic cleaning stage after each batch production.

The technical requirements are not describing since for this project, they are very similar to the functional requirements. However during dissertation some technical requirements are listed in order to explain some choices made for the hardware and software development.

[8]Confined space is an area with limited entry and egress and not suitable for human inhabitants. The maintenance of these spaces includes precautions such as as locking and tagging out connecting piping, testing of breathable air quality, forced ventilation, observation of workers in the space and a predetermined rescue plan with appropriate safety harnesses and other rescue equipment standing by

Chapter 2

Project design

The hardware and software choices and decisions for this project are explained in this chapter, highlighting the problem and the solution discovered during the studies. In the first part, the possibility to use wireless communication in health care products is studied and materials, which respect the Good Manufacturing Procedure (GMP), are analysed. After that, the hardware components are presented with their advantages and drawbacks. In the last subsection the software code is reported and explained in detail.

2.1 Physical design

In figure 2.1. and 2.2. the pilot mixers, scaled industrial mixers used for the test before the industrial production, are reported to show to reader the correct structures of the mechanical devices. For industrial secrecy reasons some parts, which are not relevant in this project, have been omitted.

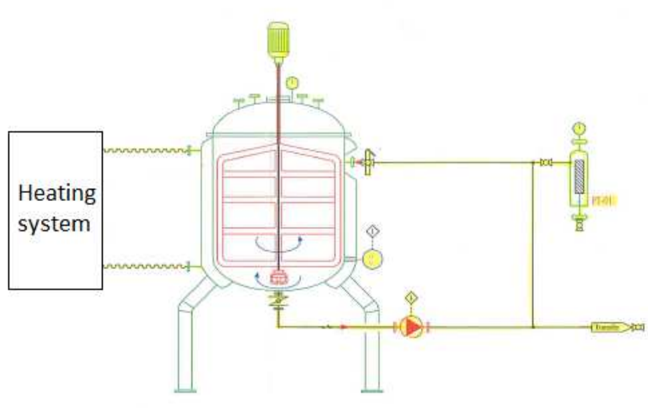

Figure 2.1. Pilot mixer 1

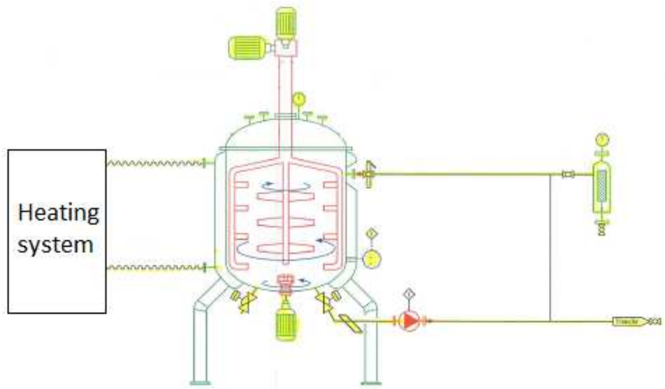

Figure 2.2. Pilot mixer 2

The main difference between the two mixers is the impeller. In the first mixer it is a traditional anchor impeller while in the second one it is made up of of an anchor, which rotates anticlockwise, and three paddles, which turn clockwise. This latter is the improved version for the emulsion products, in fact the double impeller enhances the mixing process of multi-phase products. For availability reasons, the first mixer used for the test, but the sensors can be applied also in the second mixers, without big changes in the hardware. This work leads to study the difference between heat transfer of the two types of impeller. A detail explanation on the pilot mixer used is given in the fourth chapter.
The system integrates up to nine sensors positioned in different parts of the mixer to obtain useful temperature data to use as control or for future studies. The number 9 was chosen so that there could be three sensors for each arm of the anchor, thus the temperature is measured near the centre of the mixer, in the middle and at the end of anchor arm. Since the possible arms are three the maximum number of sensors that can be used are nine. The position of the sensors allows to observe the temperature gradients in height and width with a radial symmetry. This sensors position allows to control the temperature of whole fluid in the mixer and to study and compare the data collected to the Computational Fluid Dynamics (CFD) simulations.

The two possible implementations of the sensors in the mixer are:

- wireless sensors connected to a central controller able to compute values;
- wired sensors connected to a central controller able to compute values.

The easy way to respect the GMP requirements and to avoid problems linked to the wires is the use of wireless sensors. Indeed the use of cables is critical in this

application due to the presence of movable parts described above, which can lead them being twisted, potentially damaging the whole of the system. Furthermore, the wires can lead to problems during automatic cleaning and sanitization processes of the mixer.
In the next section the transmission in fluid is studied to evaluate a possible communication able to allow the use of wireless sensors.

2.1.1 Liquid wireless transmission

In order to avoid having cables inside the industrial mixer and to respect easily the GMP requirements, the first idea is to use wireless communication between sensors and computational unit. To structure the wireless transmission, the reader needs to keep in mind that the number of sensors immersed in the liquid changes during the process due to the fact that the liquid level is not constant but it varies depending on the amount of raw materials inserted and mixed. Moreover, the walls of the vessel with the heating system piping do not allow the transmittance of a signal from the inside to the outside, thus the sensors would be able to transmit data to a controller mounted internally on the top of the mixer. Communication shall not occur in liquid but in the air or vapor, depending on the temperature. The media transmission is not homogeneous due to the presence of discontinuity liquid-air/vapor, non homogeneous of liquid until the end of mixing phase. These aspects lead different discontinuity walls to be in account in the characterization of the system

The four possible ways to transmit data through a liquid material are:

- Optical waves;
- Acoustic waves;
- Electromagnetic waves;
- Wired communication.

Optical communication uses light to transmit data in the media which can be fiber cables or free-space. As any transmission systems, it uses a transmitter, which encodes a message into a signal at an certain frequencies, in this case, optical or infrared frequencies, a channel, that is the media, which carries the signal to its destination, and a receiver, which reproduces the message from the received signal. Optical waves do not suffer from such high attenuation but are affected by scattering. Moreover, transmission of optical signals requires high precision in pointing the narrow laser beams[1]. Optical communication with use of transmission media, such as fiber, uses laser to obtain a small beam as a transmitter. Laser functionality depends strictly on the temperature and this leads to avoid the use of it in an environment with different temperature gradients. The transmitter and receiver need to be aligned and it is very difficult in a motion system where the paddles of anchor are not very flexible depending on the viscosity of the material mixed. Adding the no-homogeneity of the physical and optical properties, it leads to reject this kind of transmission which requires, during transmission phase, a current equal or greater than $200mA$.

Acoustic communication uses sound to transmit signal and it the most used kind of communication in underwater applications, particularly in the military field. The low frequency permits a long-distance transmission but with low baud-rate. Unfortunately the noise of the mixer motors with the perturbation of liquids and the large dimension of antenna make the usage of this application impossible. In addition the discontinuity between liquid and air/vapour leads to a high attenuation that can bring a loss in transmission.

The electromagnetic wireless communications are largely used in free-space transmission and it is also spreading in underwater projects, but the possible range covered depends on media conductivity. So a first analysis of conductivity, σ, is performed to have an idea about it and its variation due to the kind of final product or the temperature. The two products, an emulsion and a cleanser, are dissolved with different percentages (1%, 10%, 50%, 80% and 100%) in distilled water with a conductivity of 0,067$\frac{\mu S}{cm}$ according to the demineralized water used by the company.
The percentages of products, blend with water, are weighed using a scale with an error equal to $1g$, while the homogenization is reached mixing the samples with a rotating paddles at high speed. The measurements are taken on $250g$ of final blends. The conductivity meter used is the *CG385* by *Schott Instruments*. The error is not reported because the measurements are performed to have an idea of the conductivity and not to have the precise measurements. The temperatures tested, obtained putting the testers in the climate chamber for 8 hours, are three: 25°C, 40° and 50°C. Tables 2.1. and 2.2. report the values obtained, while figures 2.3. and 2.4. show the conductivity variations graphically.

[1] Dario Pompil, Ian F. Akyildiz, Overview of Networking Protocols for Underwater Wireless Communications, IEEE Communications Magazine, January 2009, pp 97

- Cleanser

PERCENTAGE	**T** = 25°**C [mS/cm]**	**T** = 40°**C [mS/cm]**	**T** = 50°**C [mS/cm]**
1%	0,49	0,76	0,88
10%	3,47	5,86	6,85
50%	18,6	26,8	30,8
80%	26,4	38,5	44,3
100%	31,5	45	52,7

Table 2.1. Cleanser conductivity

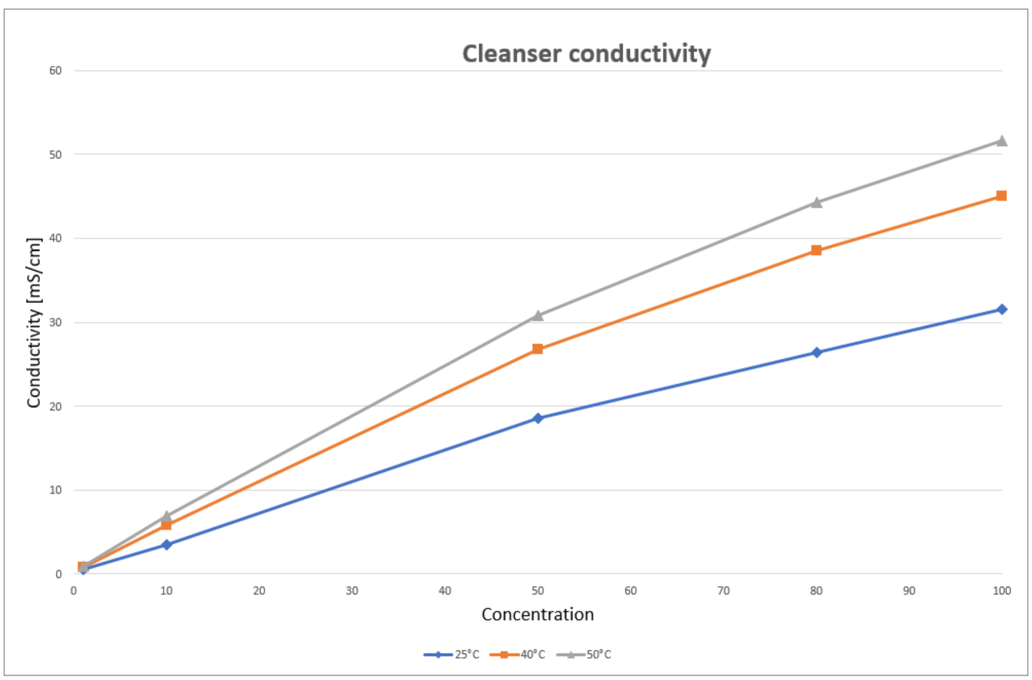

Figure 2.3. Cleaser conductivity graph

- Emulsion

PERCENTAGE	**T** = 25°**C [mS/cm]**	**T** = 40°**C [mS/cm]**	**T** = 50°**C [mS/cm]**
1%	0,04	0,06	0,08
10%	0,37	0,53	0,57
50%	1,56	2,32	2,68
80%	2,64	3,52	3,95
100%	2,77	3,85	4,67

Table 2.2. Emulsion conductivity

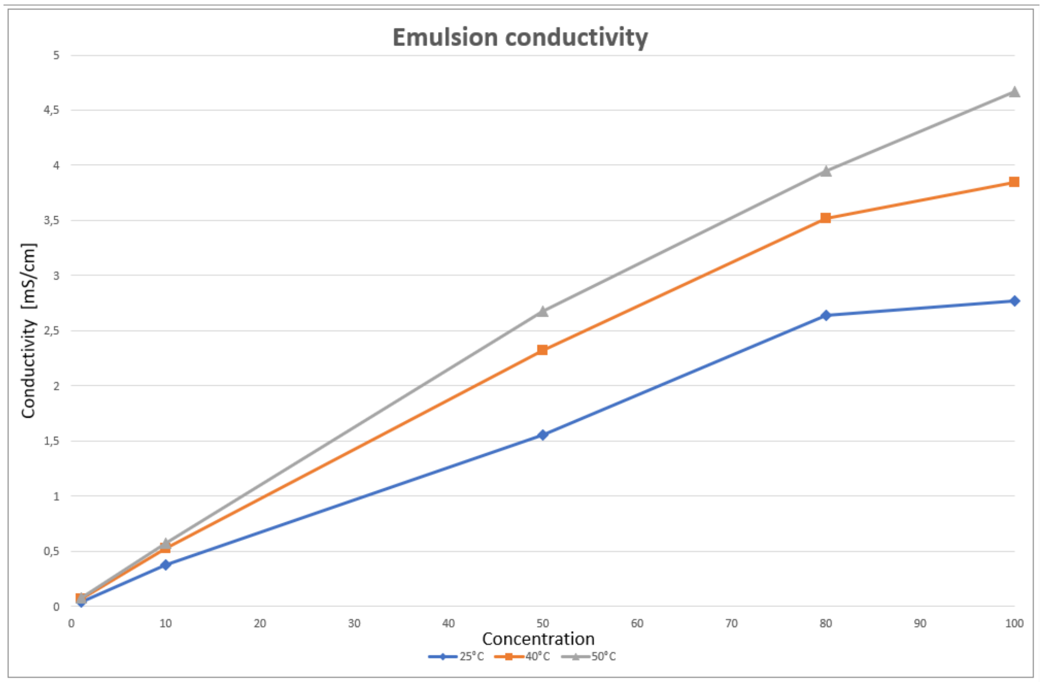

Figure 2.4. Emulsion conductivity graph

The cleanser has a very high conductivity that is higher than the marine water which has a conductivity equal to $40\frac{mS}{cm}$ and it increases linearly with the temperature.

The transmission distance of electromagnetic waves depends on the media conductivity and the frequency, the higher the frequency the shorter the distance. The common free available standard frequencies are: 868 MHz, 2.4 GHz. Therefore, the penetration depth δ (the distance where the electrical and magnetic fields are reduced of a $\frac{1}{e}$ factor) can be computed using the following formula:

$$\delta = \frac{1}{\sqrt{\pi * f * \mu * \sigma}}[m]$$

where f is the frequency of the electromagnetic wave, μ is the absolute magnetic permeability of the conductor and σ is the conductivity. While the products have high percentage of water which is a diamagnetic material, the absolute magnetic permeability can be considered the same as the vacuum magnetic permeability ($\mu_0 = 4 * \pi * 10^7$ $[\frac{H}{m}]$). The results obtained are in range from 8 to 0,2 cm, which are too low for the distances involved in the systems: from 0.5 meters for a mesh structure to few meters for a star structure.

Lower frequencies are available using RFID (Radio Frequency IDentification) technology that is employed in tag reader, as in NFC communications, and they are: Low-Frequency 125 kHz; High-Frequency 13,56 MHz, Ultra-High-Frequency 433/868 MHz; Microwave 2,45 GHz. Therefore, the penetration depth is computed for 13,56 MHz and 125 kHz and the results are reported graphically in figures 2.5. and 2.7. for the frequency 125 kHz while figures 2.6. 2.8. for the frequency 13,56 MHz.

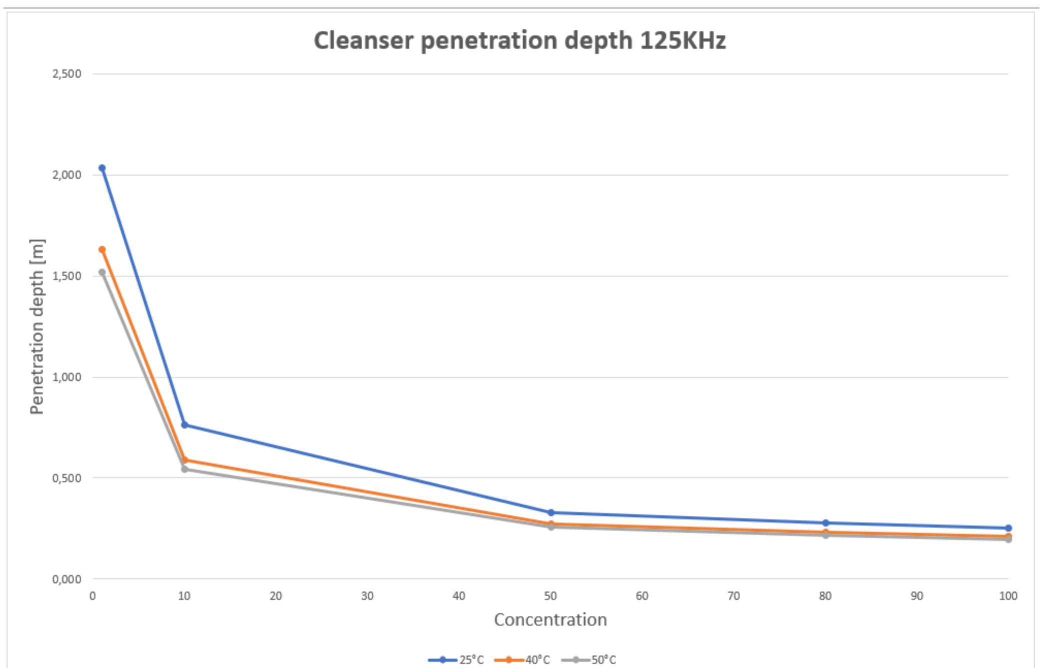

Figure 2.5. Cleanser penetration depth for 125 KHz

Figure 2.6. Cleanser penetration depth for 13,56 MHz

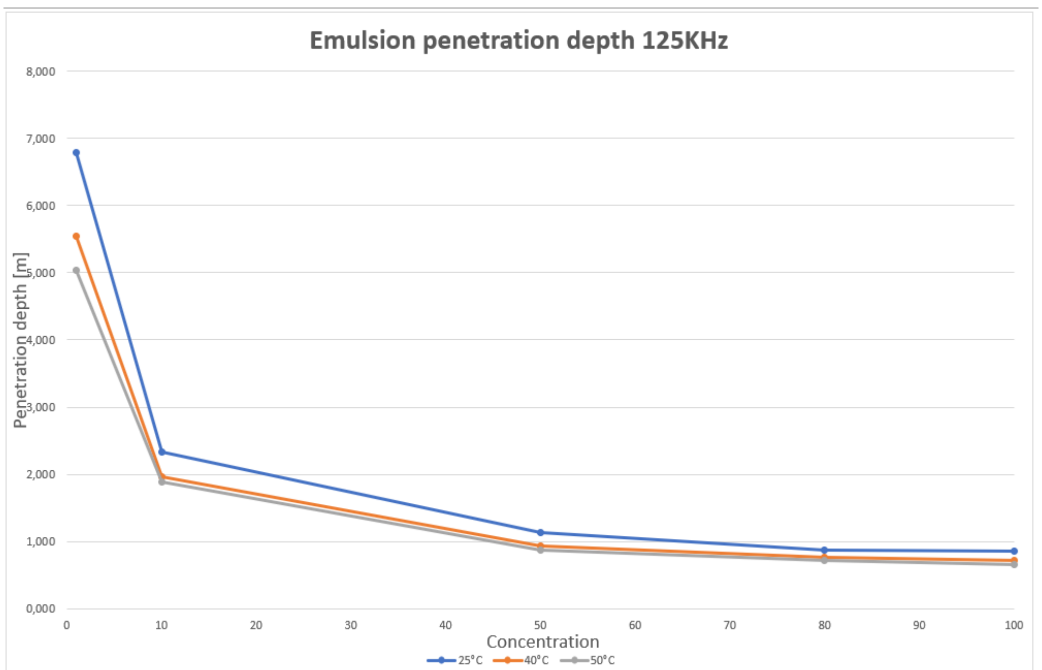

Figure 2.7. Emulsion penetration depth for 125 KHz

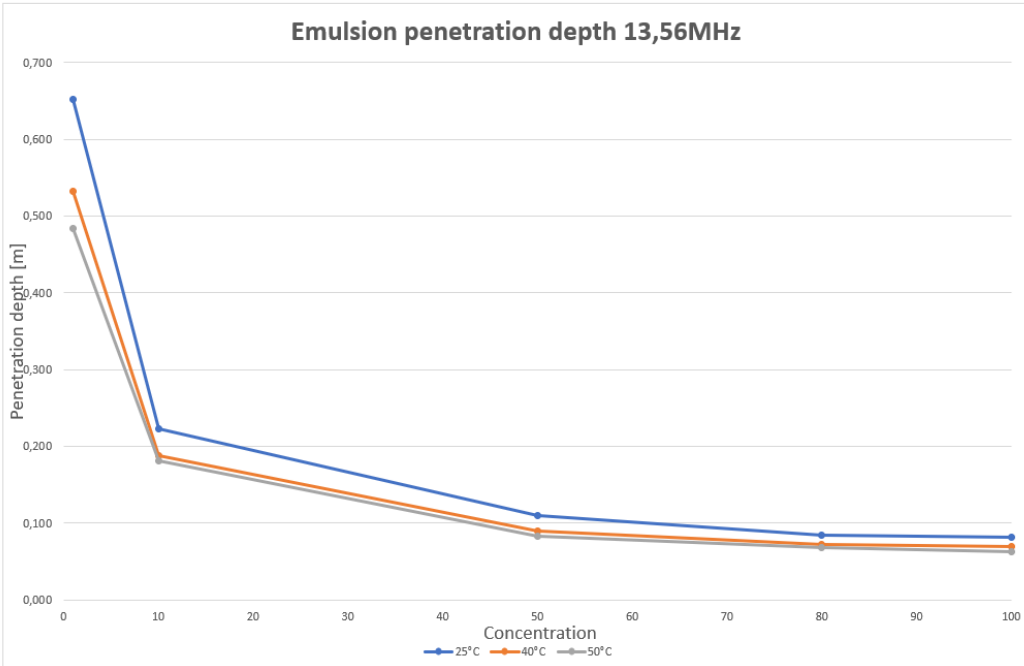

Figure 2.8.   Emulsion penetration depth for 13,56 MHz

The frequency 13,56 MHz allows too short propagation distance, so it not possible to use it, but the Low-Frequency permits enough distance with a suitable system structure. The technologies RFID at 125 KHz use the passive tags, which do not have internal power source so they are powered by the electromagnetic energy transmitted form the reader. <As the name implies, passive tags wait for a signal from an RFID reader. The reader sends energy to an antenna which converts that energy into an RF wave that is sent into the read zone. Once the tag is read within the read zone, the RFID tag's internal antenna draws in energy from the RF waves. The energy moves from the tag's antenna to the IC and powers the chip which generates a signal back to the RF system. This is called backscatter. The backscatter, or change in the electromagnetic or RF wave, is detected by the reader (via the antenna), which interprets the information[2]. The metallic structure of the mixer and impeller make the employment of this technology impossible: the antenna cannot have a conductive material below it, the dimension of it is too large and the power consumption of the reader to obtain a suitable range is too large if the device is fed with a battery.

The wired communication is the only way to connect the sensors to the controller. The cables have to respect the chemical and physical GMP requirements to avoid

[2]Suzanne Smiley, Active RFID vs. Passive RFID: What's the Difference?, https://blog.atlasrfidstore.com, March 2016

contamination in the final product. This means that only few materials are allowed and the thickness of them must be very small so as to not compromise the efficiency of propeller where the sensors are mounted.

2.1.2 Liquid wired transmission

Due to the lack of available technology to achieve the transmission in conductive fluid, the sensors will use wired transmission to communicate with the computational unit. The wired solution leads to problems linked to the GMP requirements. Therefore, the materials in contact with the product needs to be chemically compatible with the company policies. More details about the layout and design of system connection is reported in the next chapters.

The wired solution leads to a main problem linked to the motion parts. During the process, the mixer is closed and the thickness of the vessel does not allow wireless communication from the inside to the outside. This problem can be solved by bringing the signal outside the mixer by using a wire which will go through a small hole present on the top of the vessel. Later the hole shall be soldered or clamped. Thus the signal from the central computational unit can be brought outside easily but the sensors are positioned on the rotating anchor and can lead to the twisting of cables around it. To avoid this problem, the rotation part can be unmatched from fixed one using a wireless transmission, and that is possible only if the antennas of both devices are outside of the fluid during communication. It is possible because the mixer is never full up to the top. The design and position of cables and boards with their own protection cover is studied in future to respect the requirements of cleaning and sanitization phases.
A very simple schematic drawing is reported in figure 2.9, it has only a illustrative aim.

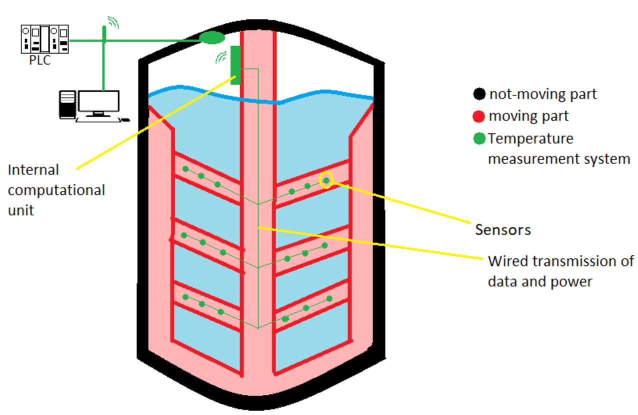

Figure 2.9. Simple schematic of system

As shown in figure 2.9. the external signal can be transmitted to a Personal Computer to display the graphically the data collected, to a PLC to control the heat system and save data in the server or through a wireless communication to external devices. This last option leads to security problems and usually is avoided in industrial plant if it is not necessary. The first solution is used during the test phases.

2.1.3 GMP Compatibility

The materials which can be used inside the mixer have to respect the good manufacturing procedure requirements. The only metal allowed is the ASIL 316L, a particular alloy described in the first chapter. It does not compromise the product and it is resistant against corrosion caused by the chemical material used in formulation of the final product.
As for the plastic materials, a guide provided by the external company Grayline™ is followed. The best material is the polytetrafluoroethylene (PTFE) and the well-known brand name of PTFE-based formulas is Teflon by Chemours.[3]. An extract of the chemical compatibility guide[4] given by Grayline™ is reported in figure 2.10, where the key is

- D = do not use at all;
- C = use with caution, may work well in limited/controlled application;
- B = some degradation;
- A = little to no effect over the entire service life.

[3] "Teflon | Chemours Teflon Nonstick Coatings and Additives". www.chemours.com. Retrieved 1 March 2016

[4] https://www.graylineinc.com/chemical-compatibility-guide.html

MATERIAL	Functional Group	Class/Use	PVC - Flexible	Polyethylene (PE)	Thermoplastic Elastomer (TPE)	Thermoplastic Rubber (TPV / Santoprene)	Fluoropolymer (PFA/ FEP/ PTFE)	Fluoroelastomer (FKM/Viton)	Rubber (Natural), Isoprene (NR, IR)	Silicone (SI)	Polyethylene terephthalate (Polyester / PET)
Acetaldehyde	Aldehyde	Precursor	C	A	A	A/B	A	C/D	B	C	-
Acetate Solvent		Acetate Solvent	D	C	C/D	B	A	D	D	D	D
Acetic Acid, 05%	Acid - Weak	Precursor	A	A/B	A	A	A	A	B	A	C
Acetic Acid, 50%	Acid - Weak	Precursor	A/C	-	-	-	A	-	-	-	D
Acetic Acid, 80%	Acid - Weak	Precursor	D	-	-	-	A	A	-	-	-
Acetone	Ketone	Solvent Polar	C	B/C	B	A	A	D	C	B	D
Alcohol - Aliphatic	Alcohol		B	A/B	A	A/B	A	A/B	A	B	B
Ammonia 25%	Base - Weak	Cleaner	B	-	-	-	A	-	-	-	-
Base - Strong	Base - Strong		B	A/C	B	A	A	A/B	A	B/D	D
Base - Weak	Base - Weak		A	-	B	A	A	D	-	A	D
Benzyl	HC Aromatic		C	A	-	C	-	A	-	C	-
Benzyl Alcohol	Alcohol / Aromatic	Solvent Borderline	B/C	-	A	A/D	A	A	D	B	D
Butanol (n-Butanol)	Alcohol	Solvent Borderline	B	A	B/D	A/B	A	A	A	B	-
Buttermilk		Food Additive	B	-	A	A	A	A	-	-	-
Calcium Carbonate (Chalk)			A	B	A	A	A	A	-	-	-
Calcium Sulfite		Food Additive	-	-	-	A	A	A	-	-	-
Carbitol	Alcohol / Ether	Solvent Borderline	C	-	-	B	A	A/B	B	B	-
Carbon Dioxide		Gas	A	B	A	B	A	A/B	B	A	-
Cinnamon Oil		Plant Oil	D	-	-	C	A	D	-	-	-
Coconut Oil (Coconut Butter)		Plant Oil	A	A	B	B	A	A	D	A	-
Dipropylene Glycol	Alcohol / Ether	Glycol	B/C	-	-	A	A	A	-	-	-
Ethanol (Ethyl Alcohol)	Alcohol	Solvent Polar	A/C	B	A/B	A/B	A	A/B	A	B	C
Ethyl Acetate	Ester	Solvent Borderline	D	C	B	A	A	D	D	B/C	D
Ethyl Chloride	HC Halogen		D	B	C	C	A	A	D	D	-
Glycerine (Glycerol)	Alcohol	Food Additive	A	A	A	A	A	A	A	A	-
Glycol		Glycol	C	B	B	A	A	A	A	B	B
Hydrobromic Acid 70-100%	Acid - Strong	Precursor	A/B	A/B	-	B	A	A	A	D	-
Hydrofluoric Acid 40-50%	Acid - Strong	Precursor	C	B	D	D	A	A	-	D	D
Hydrogen Peroxide 30%	Peroxide	Oxidizing Agent	A/B	-	B	-	A	A	-	-	B
Methanol	Alcohol	Solvent Polar	B	A	A	A	A	A/D	A	A	B
Methyl Benzoate	Ester	Solvent Borderline	-	-	-	-	A	A	-	-	-
Methyl Cellulose		Thickener	-	-	-	-	A	D	-	-	-
Milk		Food Additive	A	A/B	B	A	A	A	A	A/B	-
Mineral Oil	Petroleum	Oil	B	C	B	D	A	A	D	B	B
Nitric Acid 50%	Acid - Strong	Precursor	C	C	C	C	A	A	-	C	B
Olive Oil	Fatty Acids		C	A	D	B	A	A	D	C	-
Palm Oil		Plant Oil	C	-	-	B	A	A	-	-	-
Paraffin	HC Aliphatic		B	-	D	A	A	A	-	-	-
Potassium Acetate	Ester	Flavoring	A	-	A	A	A	D	A	D	-
Potassium Hydroxide 30%	Base - Strong	Precursor	A	-	A	-	A	-	-	-	-
Propylene Glycol	Alcohol	Glycol	B/C	A/B	A	A	A	A	-	-	-
Propylene Oxide	Ether	Precursor	C	B	A	A	A	D	D	D	-
Salt Solutions, Metallic		Salt	A	-	-	-	A	-	-	-	-
Sodium Bicarbonate			A	B	B	A	A	A	A	A/C	-
Sodium Carbonate		Water Treatment	A	B	A	A/B	A	A	A	A	B
Sulfuric Acid 10-75%	Acid - Strong	Oxidizing Agent	A/B	B	B	A/B	A	A	-	D	A/B

Figure 2.10. Extract of chemical compatibility table

2.2 Hardware: components and connection

Following figure 2.9, the main parts of the system are the computational units (transmitter and receiver) and the temperature sensors. In this section the hardware is analysed in detail highlighting the choices made and the possible future steps to improve the hardware to optimize the entire system.

2.2.1 Temperature sensor

The temperature sensors usually use three methods to measure heat quantity, described as follows:

- Seebeck effect: when two wires of dissimilar metals are joined at one end and this end is heated, a thermoelectric voltage arises across the circuit. This method is used in thermocouples and it allows to measure of a large range of temperatures with a fast response at low cost. The main disadvantages are linked to the reference junction which has to be arranged and the strongly not linear calibration function. The voltages produced by seedbeck effect are small, usually only a few microvolts per degree of temperature difference at the junction. If it is large enough, some device based on this principle can produce a few millivolts[5].

- Resistive temperature detector (RTD): the principle is based on the dependence of the electrical resistance of metals on the temperature. Depending on the material a high stability and linearity on a large range can be establish with a high resistivity but also an high cost and high time constant. Most RTD elements consist of a length of fine coiled wire wrapped around a ceramic or glass core[6]. The main drawbacks of these sensors are the fragility of the element and the resistive output. The electrical resistance of which changes with temperature as approximated by this formula: $R_T = R_{ref}[1+\alpha(T-T_{ref})]$ Typical sensors based on this principle are PT100 and PT1000.

- Silicon band gap: these sensors are based on the principle that the forward voltage drop of a silicon diode is temperature dependent. By measuring the forward voltage drop across the diode, the temperature of the silicon is computed. Indeed the energy bandgap of semiconductor tends to decrease as the temperature is increased. This behaviour can be better understood if one considers that the interatomic spacing increases when the amplitude of the atomic vibrations increases due to the increased thermal energy. This effect is quantified by the linear expansion coefficient of a material. An increased interatomic spacing decreases the potential seen by the electrons in the material, which in turn reduces the size of the energy bandgap. A direct modulation of the interatomic distance, such as by applying high compressive (tensile) stress, also causes an increase (decrease) of the bandgap.[7] The bandgap energy follows this formula: $E_g(T) = E_g(0) - \frac{\alpha T^2}{T+\beta}$. The main disadvantage is the non-linearity of produced voltage with respect to te temperature. A typical sensor is the DS18B20.

From the above list, the first element can be excluded since the thermocouples need two different temperatures at the two ends of the junction, and it is not possible in this project because in some case the temperature in the whole mixer is the same, therefore no voltage is produced by the sensors.

[5]https://searchnetworking.techtarget.com/definition/Seebeck-effect
[6]https://in.omega.com/prodinfo/rtd.html
[7]Principle of Semiconductor Devices, B. Van Zeghbroeck, 2011

The resistive temperature sensors are highly accurate but have a bad time response. These sensors can be analog or digital. The analog sensors require a Wheatstone bridge for each sensor to respect the requirements about accuracy and this means another hardware which has to protect from chemical, temperature and moisture damages and in addition it needs an industrial calibration. This leads to higher costs and more time. To avoid all these issues, the digital sensor can be used if it is calibrated by the producer or seller. Unfortunately all commercial digital RTD sensors are not waterproof nor chemical compatible so they need a protection for each sensor.

The last class of sensors are sold in a waterproof digital configuration with the calibration carried out by the producer. This solves the of the non-linearity present in formula, indeed the device can compute the right temperature thanks to its embedded computational unit. One of these sensors is *642 by Adafruit*. This is a pre-wired and waterproofed version of the DS18B20 sensor made with a PTFE wire cable. The PTFE is one of the few materials resistant until the 220°C and chemically respects GMP requirements. The sensor is encapsulated in a stainless steel 316 tube so it is not recommended for salt-water or corrosive environments, but it can be changed with stainless steel 316L, the same material of the mixer tank.
The physical specifications are:

- Stainless steel 316 tube 6 mm diameter approximately 30 mm long;
- A cable approximately 90 cm long;
- It contains DS18B20 temperature sensor;
- Three wires: orange one connects to 3-5 V, white connects to ground and blue one is data line.

The integrated *DS18B20* sensor is a digital thermometer by Maxim that provides 9-bit to 12-bit Celsius temperature measurements and has an alarm function with nonvolatile user-programmable upper and lower trigger points. It communicates over a 1-wire bus that by definition requires only one data line for the communication of data with a central microprocessor. Each sensor has a unique 64-bit serial code, which allows multiple DS18B20s to function on the same 1-wire bus. Thus, it is simple to use one microprocessor to control many sensors distributed over a large area. Its temperature range is form $-55°C$ to $125°C$ and power supply contains between 3 to 5 V. Figure 2.11. shows the block diagram of DS18B20.

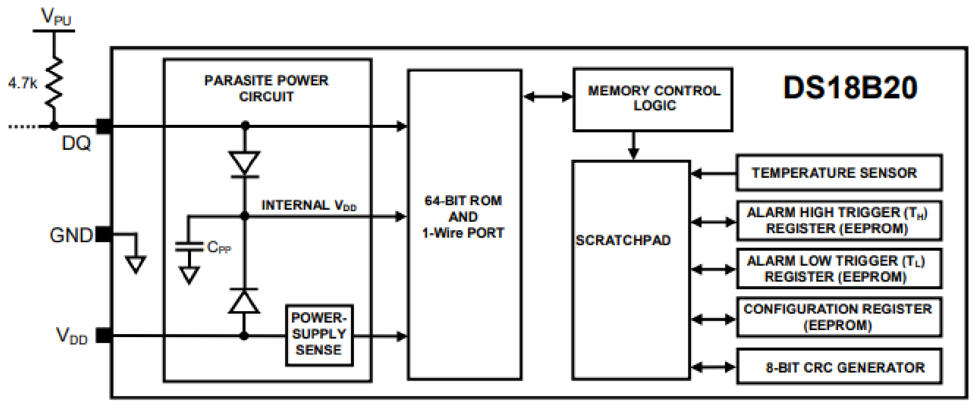

Figure 2.11. DS18B20 block diagram

The 1-wire protocol and bus allow to avoid more than three wires in the mixer, indeed the sensors can be connected together in a parallel way, so only three wires are needed: two for power supply and one for the digital signal. This aspect simplifies the respect of GMP requirements. A simple scheme to understand the connection is reported in figure 2.12.

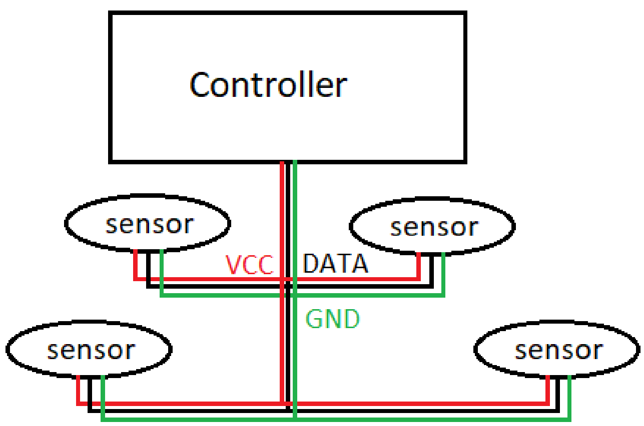

Figure 2.12. Simple example of connection

The main characteristics are listed in figure 2.13, from the vendor datasheet.

AC ELECTRICAL CHARACTERISTICS—NV MEMORY

(-55°C to +100°C; V_{DD} = 3.0V to 5.5V)

PARAMETER	SYMBOL	CONDITIONS	MIN	TYP	MAX	UNITS
NV Write Cycle Time	t_{WR}			2	10	ms
EEPROM Writes	N_{EEWR}	-55°C to +55°C	50k			writes
EEPROM Data Retention	t_{EEDR}	-55°C to +55°C	10			years

AC ELECTRICAL CHARACTERISTICS (-55°C to +125°C; V_{DD} = 3.0V to 5.5V)

PARAMETER	SYMBOL	CONDITIONS	MIN	TYP	MAX	UNITS	NOTES
Temperature Conversion Time	t_{CONV}	9-bit resolution			93.75	ms	1
		10-bit resolution			187.5		
		11-bit resolution			375		
		12-bit resolution			750		
Time to Strong Pullup On	t_{SPON}	Start Convert T Command Issued			10	µs	
Time Slot	t_{SLOT}		60		120	µs	1
Recovery Time	t_{REC}		1			µs	1
Write 0 Low Time	t_{LOW0}		60		120	µs	1
Write 1 Low Time	t_{LOW1}		1		15	µs	1
Read Data Valid	t_{RDV}				15	µs	1
Reset Time High	t_{RSTH}		480			µs	1
Reset Time Low	t_{RSTL}		480			µs	1,2
Presence-Detect High	t_{PDHIGH}		15		60	µs	1
Presence-Detect Low	t_{PDLOW}		60		240	µs	1
Capacitance	$C_{IN/OUT}$				25	pF	

DC ELECTRICAL CHARACTERISTICS (-55°C to +125°C; V_{DD}=3.0V to 5.5V)

PARAMETER	SYMBOL	CONDITIONS	MIN	TYP	MAX	UNITS	NOTES
Supply Voltage	V_{DD}	Local Power	+3.0		+5.5	V	1
Pullup Supply Voltage	V_{PU}	Parasite Power	+3.0		+5.5	V	1,2
		Local Power	+3.0		V_{DD}		
Thermometer Error	t_{ERR}	-10°C to +85°C			±0.5	°C	3
		-55°C to +125°C			±2		
Input Logic-Low	V_{IL}		-0.3		+0.8	V	1,4,5
Input Logic-High	V_{IH}	Local Power	+2.2		The lower of 5.5 or V_{DD} + 0.3	V	1, 6
		Parasite Power	+3.0				
Sink Current	I_L	$V_{I/O}$ = 0.4V	4.0			mA	1
Standby Current	I_{DDS}			750	1000	nA	7,8
Active Current	I_{DD}	V_{DD} = 5V		1	1.5	mA	9
DQ Input Current	I_{DQ}			5		µA	10
Drift				±0.2		°C	11

Figure 2.13. DS18B20 electrical characteristics

2.2.2 Controller

The controllers are in charge of computing and transmitting the data collected by the sensors. To avoid high costs of design and realization, the microcontroller boards are used, to have, also, in future the possibility to add sensors or other devices without changes. The mounted boards used are realized by Arduino™, an Italian open-source hardware and software company, project and user community that designs and manufactures excellent single-board microcontrollers. Its products are licensed under the GNU Lesser General Public License (LGPL) or the GNU General Public License (GPL), this allows freedom in the usage and modification. The same company also provides Arduino Integrated Development Environment (IDE) which is a cross-platform application that permits to program the boards supporting C and C++ program languages using special rules of code structuring[8].

The best suitable Arduino™ board is the MKR WAN 1300, shown in figure 2.14. It has been designed to offer a pratical and cost effective solution for makers seeking to add LoRa® connectivity to their projects.

Figure 2.14. Arduino MKR WAN 1300

LoRa® is the physical layer or the wireless modulation utilized to create the long range communication link. Many legacy wireless systems use frequency shifting keying (FSK) modulation as the physical layer while LoRa® is based on chirp spread spectrum modulation, which has lower power consumption than FSK modulation and it significantly increases the communication range. Chirp spread spectrum has been used in military and space communication for decades due to the long communication distances that can be achieved and robustness to interference, but LoRa® is the first low cost implementation for commercial usage. Wireless communication

[8]https://www.arduino.cc/en/guide/introduction

based on LoRa® protocol defines ten channels with a bandwidth of 125/250 kHz in the frequency band 867-869 MHz for the Europe. The maximum power in transmission is +14dBm with a data rate included in the range 250bps - 50kbps.[9]

The board is based on the *Atmel SAMD21* microcontroller and a *Murata CMWX1ZZABZ* LoRa® module. The board has the ability to be powered using two 1.5V AA or AAA batteries or external 5V, switching from one source to the other automatically. The circuit operating voltage is equal to 3.3V and the board has 8 digital I/O pins, 7 analog input pins and 1 analog output pin. The most important specifications are reported in table 2.3.

Microcontroller	SAMD21 Cortex-M0+
Radio Module	CMWX1ZZABZ
Temperature range	0-85°C
Board Power Supply (USB/VIN)	5 V
Supported Batteries	2x AA or AAA
Circuit Operating Voltage	3.3 V
Digital I/O Pins	8
PWM Pins	12 (0,1,2,3,4,5,6,7,8,10,A3 or 18, A4 or 19)
Protocol	UART, SPI, I2C
Analog Input Pins	7 (ADC 8/10/12 bit)
Analog Output Pins	1 (DAC 10 bit)
DC Current per I/O Pin	7 mA
Flash Memory	256 KB
SRAM	32KB
Clock Speed	32.768 KHz (RTC), 48 MHz
Led Builtin	6
Antenna power	2 dB
Carrier frequency	868/915 MHz
Length	67.64 mm
Width	25 mm
Weight	32 gr

Table 2.3. Arduino MKR WAN 1300 specifications

As reported in the table the dimensions are small ($67.64x25x20mm$) and it allows an easy implementation in the mixer.

[9]LoRa Alliance, A technical overview of LoRa® and LoRaWAN™, 2015, pp 12

The Atmel SAMD21 is a low-power microcontroller using the 32-bit ARM Cortex-M0+ processor, ranging 48-pins with 256 KB Flash and 32 KB of SRAM. It can operate at a maximum frequency of 48 MHz, and it is designed for simple and intuitive migration with identical peripheral modules, hex compatible code, identical linear address map, and pin compatible migration paths between all devices in the product series.
All integrated devices have accurate and low-power external and internal oscillators. All oscillators can be used as a source for the system clock. Different clock domains can be independently configured to run at different frequencies, enabling power saving by running each peripheral at its optimal clock frequency, and thus maintaining a high CPU frequency while reducing power consumption.[10]
The SAMD21 has two software-selectable sleep modes, "Idle" and "Stand-by". In the Idle mode, the CPU is stopped while all other functions can be kept running. In Stand-by mode, all clocks and functions are stopped, except those selected to continue running. The figure 2.15. reports the Atmel SAMD21 specifications.

The CMWX1ZZABZ by Murata is a compact, low cost and low power wide area network (LPWAN) wireless module that supports, as said previously, the LoRaWAN long range wireless protocol. Communication with this module can be achieved via UART, SPI or I2C peripheral interfaces. An ADC and up to 18 GPIOs provide plenty of flexibility for connecting sensors, switches and status leds, and it is powered from a 2.2 to 3.6 VDC supply. The module has pre-certified radio regulatory approvals for operating in the 868 and 915 MHz industrial, scientific and medical (ISM) spectrum in most geographical regions of the world. The normal output power is +14 dBm but a PA boost function can be selected to increase RF output to +20dBm for long range applications or those where the end-device is sited in a poor signal location indoors. The MCU includes 192 kB flash and 20 kB RAM, and has enough memory to embed customer applications and host other modulation stacks.[11]

[10] Atmel SAMD21 datasheet
[11] Murata CMWX1ZZABZ datasheet

- Processor
  - ARM Cortex-M0+ CPU running at up to 48MHz
    - Single-cycle hardware multiplier
    - Micro Trace Buffer (MTB)
- Memories
  - 32/64/128/256KB in-system self-programmable Flash
  - 4/8/16/32KB SRAM Memory
- System
  - Power-on reset (POR) and brown-out detection (BOD)
  - Internal and external clock options with 48MHz Digital Frequency Locked Loop (DFLL48M) and 48MHz to 96MHz Fractional Digital Phase Locked Loop (FDPLL96M)
  - External Interrupt Controller (EIC)
  - 16 external interrupts
  - One non-maskable interrupt
  - Two-pin Serial Wire Debug (SWD) programming, test and debugging interface
- Low Power
  - Idle and standby sleep modes
  - SleepWalking peripherals
- Peripherals
  - 12-channel Direct Memory Access Controller (DMAC)
  - 12-channel Event System
  - Up to five 16-bit Timer/Counters (TC), configurable as either:
    - One 16-bit TC with compare/capture channels
    - One 8-bit TC with compare/capture channels
    - One 32-bit TC with compare/capture channels, by using two TCs
  - Three 24-bit Timer/Counters for Control (TCC), with extended functions:
    - Up to four compare channels with optional complementary output
    - Generation of synchronized pulse width modulation (PWM) pattern across port pins
    - Deterministic fault protection, fast decay and configurable dead-time between complementary output
    - Dithering that increase resolution with up to 5 bit and reduce quantization error
  - 32-bit Real Time Counter (RTC) with clock/calendar function
  - Watchdog Timer (WDT)
  - CRC-32 generator
  - One full-speed (12Mbps) Universal Serial Bus (USB) 2.0 interface
    - Embedded host and device function
    - Eight endpoints
  - Up to six Serial Communication Interfaces (SERCOM), each configurable to operate as either:
    - USART with full-duplex and single-wire half-duplex configuration
    - I^2C up to 3.4MHz
    - SPI
    - LIN slave
  - One two-channel Inter-IC Sound (I^2S) interface
  - One 12-bit, 350ksps Analog-to-Digital Converter (ADC) with up to 20 channels
    - Differential and single-ended input
    - 1/2x to 16x programmable gain stage
    - Automatic offset and gain error compensation
    - Oversampling and decimation in hardware to support 13-, 14-, 15- or 16-bit resolution
  - 10-bit, 350ksps Digital-to-Analog Converter (DAC)
  - Two Analog Comparators (AC) with window compare function
  - Peripheral Touch Controller (PTC)
    - 256-Channel capacitive touch and proximity sensing
- I/O
  - Up to 52 programmable I/O pins
- Drop in compatible with SAM D20
- Packages
  - 64-pin TQFP, QFN, UFBGA
  - 48-pin TQFP, QFN, WLCSP
  - 32-pin TQFP, QFN, WLCSP
- Operating Voltage
  - 1.62V – 3.63V

Figure 2.15. Atmel SAMD21 specifications

2.2.3 Antenna 868 MHz

Many different antennas are commercially available and they can be divided in two main groups: printed circuit or rigid antennas. These groups have two big differences:

- flexibility: the printed circuit antennas are very thin and flexible and this allows a better integration in the mixer, paying attention on the metallic surface under it.
- gain: the rigid antennas have a higher peak gain that allows longer transmission distance in particular in a moist environment.

The chosen antenna, with UFL as connector compatible with Arduino MKR WAN 1300 is the *105262-0002* by Molex, as printed circuit antenna. The main characteristics are reported in the table 2.4.

Type	Printed Circuit
Bands(MHz)	863-870 902-928
Peak gain(dBi)	1.3
Polarization	linear
Cable length(mm)	150.00
Connector	UFL
Length(mm)	79.00
Width(mm)	10.00

Table 2.4. Antenna technical caracteristics

2.2.4 Tilt sensor

Tilt sensors allow to detect orientation or inclination. This kind of sensors are small, inexpensive, low-power and easy-to-use. Their simplicity makes them popular for toys, gadgets and appliances. Sometimes they are referred to as "mercury switches", "tilt switches" or "rolling ball sensors" for design reasons.
They are usually made by a cavity of some sort (cylindrical is popular, although not always) and a conductive free mass inside, such as a blob of mercury or rolling ball. One end of the cavity has two conductive elements (poles). When the sensor is oriented so that that end is downwards, the mass rolls onto the poles and shorts them, acting as a switch throw.[12]
While not as precise or flexible as a full accelerometer, tilt switches can detect motion or orientation in a simple way. Another advantage to them is that the big ones can

[12]https://learn.adafruit.com/tilt-sensor/overview

switch power on their own. Accelerometers, on the other hand, output digital or analog voltage that must then be analysed with extra circuitry.
So, since our board needs to acquire temperature during a mixing process and not when the mixer is not used for a process, the tilt sensors are connected to the internal mixer board to detect if the anchor is rotating or not, that means mixing procedure is running. The low power consumption and the need to not know the correct motion of the anchor makes the tilt sensor the best suitable choice.

2.3 Software implementation

In this section the code implemented in the both Arduino boards is shown and explained in detail.

2.3.1 Address code

This code is used to set the ID number of each sensor in an ascending order so that it becomes possible to list the temperatures acquired, in accordance with the ID, as shown in the sender code.

```
//Libraries
#include <OneWire.h>
#include <DallasTemperature.h>

// One Wire setting
#define ONE_WIRE_BUS      3
OneWire oneWire(ONE_WIRE_BUS);
DallasTemperature sensors(&oneWire);

//Global variables
uint8_t deviceCount = 0;
float Temp;
int id = 0 ;

//printAddress function
void printAddress(DeviceAddress deviceAddress)
{
  for (uint8_t i = 0; i < 8; i++)
  {
    // zero pad the address if necessary
    if (deviceAddress[i] < 16) Serial.print("0");
    Serial.print(deviceAddress[i], HEX);
```

```
  }
}

void setup(void)
{
  Serial.begin(9600);
  while (!Serial.available()){ //wait the serial
    ;
  }
  Serial.println(__FILE__);
  Serial.println("Write␣user␣ID␣to␣DS18B20\n");

  sensors.begin();

  // count devices
  deviceCount = sensors.getDeviceCount();
  Serial.print("#devices:␣");
  Serial.println(deviceCount);

  //Print IDs of devices
  Serial.println();
  Serial.println("current␣ID's");
  for (uint8_t index = 0; index < deviceCount; index++)
  {
    DeviceAddress t;
    sensors.getAddress(t, index);
    printAddress(t);
    Serial.print("\t\tID:␣");
    int id = sensors.getUserData(t);
    Serial.println(id);
  }

  //Enter the new IDs
  Serial.println();
  Serial.print("Enter␣ID␣for␣batch:␣");
  int c = 0;
  while (c != '\n' && c != '\r')
  {
    c = Serial.read();
    switch(c)
    {
```

```
    case '0'...'9':
      id *= 10;
      id += (c - '0');
      break;
    default:
      break;
    }
  }
  Serial.println();
  Serial.println(id);
  Serial.println();

  //Change the IDs
  Serial.println("Start labeling ...");
  for (uint8_t index = 0; index < deviceCount; index++)
  {
    Serial.print(".");
    DeviceAddress t;
    sensors.getAddress(t, index);
    sensors.setUserData(t, id);
  }
  Serial.println();

  //Print the new IDs
  Serial.println();
  Serial.println("Show results ...");
  for (uint8_t index = 0; index < deviceCount; index++)
  {
    DeviceAddress t;
    sensors.getAddress(t, index);
    printAddress(t);
    Serial.print("\t\tID: ");
    int id = sensors.getUserData(t);
    Serial.println(id);
  }
  Serial.println("Done ...");
}

void loop(void) {
}
```

- **Line 1 - 13:** In this first part the libraries are set up, the OneWire protocol is initialized and the global variables are listed.

- **Line 15 - 24:** The function Print Address () is introduced to allow the printing of the device address on the peripheral display.

- **Line 26 - 35:** First of all, in the *void setup()* function, the serial port is set up with the standard baudrate equal to 9600 bits/sec. Therefore the program waits for the user to open the terminal in order to continue. This is possible thanks to the instruction *while(Serial.available(){})*. After that the sensor are initialized.

- **Line 37 - 40:** The number of devices connected to the pin set for the OneWire protocol is printed. This is used to check the connection of the sensor.

- **Line 42 - 53:** The ID numbers of the devices detected is printed on the terminal.

- **Line 55 - 74:** The program waits that user entered the new numbers of ID for the devices checking if the characters entered are numbers.

- **Line 76 - 85:** The devices are labelled with the new IDs entered in the previous step.

- **Line 87 - 100:** The updated information of devices is printed, so the user can check the correctness of the result.

- **Line 102 - 103:** No recursive function is used in this case, therefore the **void loop()** function is empty.

This program is used for each sensor individually to set the ID to have a correlation between the temperature read and the sensor, as is explained in the next subsection. This leads to know which sensor measures a specific temperature. An example of program is shown in the figure 2.16.

Figure 2.16. Address code terminal

2.3.2 Sender code

This is the code implemented in the computational unit mounted internally in the mixer on the moving part and connected directly to the temperature sensors.

```
//Libraries
#include <SPI.h>
#include <LoRa.h>
#include <OneWire.h>
#include <DallasTemperature.h>
#include <ArduinoLowPower.h>

//One Wire settings
// Pin where data-line of temperature sensors are connected
#define sensor_pin 3
// Setup a onewire instance to communicate with any onewire devices
OneWire oneWire(sensor_pin);
// Pass oneWire reference to Dallas Temperature
DallasTemperature sensors(&oneWire);

// Global variables
int N = 9; //# of sensors
float Temp[9];
int i, id, y;
int old_vib1, old_vib2, old_vib3;
```

```
int vib1, vib2, vib3;
bool sem;
DeviceAddress t;

void setup() {
  Serial.begin(9600);

  while (!LoRa.begin(868E6)) {
    delay(2000);
  }

  sem = false;
  y = 0;

  sensors.begin();
  sensors.setResolution(12);

  delay(1000);
}

void loop() {

  //If no motion is detected
  while(not sem){
    digitalWrite(1,HIGH);
    vib1 = analogRead(A5);
    vib2 = analogRead(A4);
    vib3 = analogRead(A3);
    digitalWrite(1, LOW);
    if(((vib1 > old_vib1+15) or (vib1 < old_vib1-15)) or ((vib2 > old_vib2+15) or
    (vib2 < old_vib2-15)) or ((vib3 > old_vib3+15) or (vib3 < old_vib3-15))){
      sem = true;
      old_vib1 = vib1;
      old_vib2 = vib2;
      old_vib3 = vib3;
    }else{
      sem = false;
     }
    LowPower.deepSleep(60000); //60 seconds
```

```
}

//Check the presence of motion
if(y == 360){ //30 minutes
  y = 0;
  digitalWrite(1,HIGH);
  vib1 = analogRead(A5);
  vib2 = analogRead(A4);
  vib3 = analogRead(A3);
  digitalWrite(1, LOW);
  if(((vib1 > old_vib1+15) or (vib1 < old_vib1-15)) or ((
     vib2 > old_vib2+15) or
  (vib2 < old_vib2-15)) or ((vib3 > old_vib3+15) or (vib3
      < old_vib3-15))){
    sem = true;
    old_vib1 = vib1;
    old_vib2 = vib2;
    old_vib3 = vib3;
  }else{
     sem = false;
   }
}

//Perform temperature measurements
if(sem) {
 sensors.requestTemperatures();
 for (i=0; i<N; i++) {
   sensors.getAddress(t, i);
   id = sensors.getUserData(t);
   Temp[id-1] = sensors.getTempCByIndex(i);
 }

 //LoRa send
 LoRa.beginPacket();
 for(i=0; i<N-1; i++){
  LoRa.print(Temp[i]);
  LoRa.print(";");
 }
 LoRa.println(Temp[N]);
 LoRa.endPacket();
}
```

```
99    LowPower.deepSleep(30000);
100   y = y + 1;
101 }
```

- **Line 1 - 6:** The libraries used are included in the code. *SPI.h* and *OneWire.h* are used to communicate with the temperature sensors. *Lora.h* to communicate using LoRa protocol while *DallasTemperature.h* to use the commands to perform the temperature reading with DS18B20 sensors. The last library *ArduinoLowPower.h* is used to move the board in sleep state to save power.

- **Line 8 - 14:** In this block the pin for the OneWire communication is set up and the protocol also is set.

- **Line 16 - 23:** The global variables are initialized in order to use them in the program.

- **Line 25 - 39:** This is the *void setup()* function, in which the communications is set up. *Serial.begin()* is used to set the baudrate of the serial interface, while *LoRa.begin()* initializes the wireless communication based on LoRa protocol. The parameter 868E6 refers to the band used, the European channel centered in 868 MHz. *Sensors.begin()* initializes the sensors and then with the *sensors.setResolution(12)* the internally analog-digital converter in each sensors is set to 12 bit, the maximum possible.

- **Line 41 - 60:** This the first part of function *void loop()* where a recursive function is implemented in the board. The *while(not sem)* is a loop activated when the *sem* variable is low, that is when no motion is captured by the sensors. The digital pin 1 goes to high state in order to power the sensors and the voltages of the three sensors are acquired and converted by the internal analog-digital converter in the Arduino board. Then a comparison is made, if the result is not equal to the old one (+/- 15 to avoid a false signal for a small vibration) the *sem* variable became true, so the anchor is in motion, otherwise the *sem* variable remains false. This loop is introduced to save power, indeed the temperature is not acquired until the anchor is stopped, which means that no mixing process is started. This verification is processed every 60 seconds, a reasonable time to not miss important data. In the 60 seconds in which the board wait, it goes to the *deepSleep()* to improve the power saving.

- **Line 62 - 79:** This *if()* statement allows to control if the anchor is in motion every 30 minutes and it works similarly to the previous *while()* loop. The anchor rotates for all mixing process, so when it is stopped, no mixing procedure is running. The program waits 30 minutes before interrupting temperature acquisition in order to observe any changes in the temperature until the mixer is emptied , which usually occurs 20 minutes after the anchor stops.

- **Line 81 - 88:** In this block, if the *sem* variable is high, the temperature is acquired by each sensor by the function *sensors.requestTemperatures()* and it is with the function *sensors.getTempCByIndex()* that we are able to read the temperatures acquired by the sensors. With the *for()* statement the temperatures are saved and listed in an array according to their ID number to know the correlation between the temperatures and the sensors.

- **Line 90 - 102:** In this last part of the *void loop()* function the temperatures are sent wireless to the second controller using the functions: *LoRa.beginPacket()* which initializes the packet, *LoRa.print()* which send the object of function and *LoRa.endPacket()* that terminates the packet. After the transmission the board goes to the *deepSleep* state for 30 seconds according to the specification of the project, measuring the temperature every 30 seconds.

2.3.3 Receiver code

This is the code implemented in the computational unit which receives the temperatures and brings them outside the vessel. This code is related to the *PLX DAQ* explained in the next subsection, indeed many instruction are linked with a Excel macro installed with the program.

```
//Libraries
#include <SPI.h>
#include <LoRa.h>

//Global variables
float Temp[9];
int i;
int led =  LED_BUILTIN;;
bool boolread, boolsave;

void setup() {
  Serial.begin(9600);
  Serial.println("CLEARSHEET");
  Serial.println("LABEL, Date, Time, Temp1, Temp2, Temp3, Temp4,
  Temp5, Temp6, Temp7, Temp8, Temp9");

```

```
17   while (!LoRa.begin(868E6)) {
18     Serial.println("Starting␣LoRa␣failed!");
19     delay(2000);
20   }
21 }
22
23 void loop() {
24   Serial.println("CUSTOMBOX1,LABEL,Read␣data?");
25   Serial.println("CUSTOMBOX2,LABEL,Save␣data?");
26   Serial.println("CUSTOMBOX1,GET");
27   boolread = Serial.readStringUntil(10).toInt();
28   Serial.println("CUSTOMBOX2,GET");
29   boolsave = Serial.readStringUntil(10).toInt();
30
31   //Read procedure
32   if(boolread == true) {
33     // try to parse packet
34     int packetSize = LoRa.parsePacket();
35     if (packetSize) {
36       Serial.print("DATA,DATE,TIME,");
37       // read packet
38      if (LoRa.available()) {
39       for (i=0; i<(packetSize-2); i++) {
40         Serial.print((char)LoRa.read());
41       }
42       Serial.println((char)LoRa.read());
43      }
44    }
45   }
46
47   //Save procedure
48   if(boolsave == true) {
49     Serial.println("SAVEWORKBOOKAS,data_save");
50   }
51 }
```

- **Line 1 - 9:** In this first part of program the libraries are initialized as the global variables.
- **Line 11 - 21:** In the *void setup()* function the serial port is set up and the label of the file excel is written. Then the LoRa communication in initialized.

- **Line 23 - 29:** The first and second option as shown in figure 2.19 is set up and read with the functions *Serial.println("CUSTOMBOXx, GET")*.
- **Line 31 - 45:** If the option "Read data?" is check on the control panel in the Excel file, the board is ready to receive the data from the sender board. The variable packSize is used to save the the byte of the packet received. The function *LoRa.read()* read a byte sent by the other board.
- **Line 47 - 51:** In this last part of the program, if the option "Save data?" is checked, the Excel file is saved.

2.3.4 PLX DAQ v2

The receiver code is strictly connected to the tool *PLX DAQ v2* which is a program used to establish an easy communication between Microsoft Excel on a Windows Computer and any device that supports serial port protocol. Indeed it was intentionally written to allow communication between Arduino and Excel[13].
After the installation of the new software, the "Simple Data" sheet can be open with Excel. In this file it is possible to visualize the control panel from where it is possible to connect the device and adjust settings. This is reported in figure 2.17.

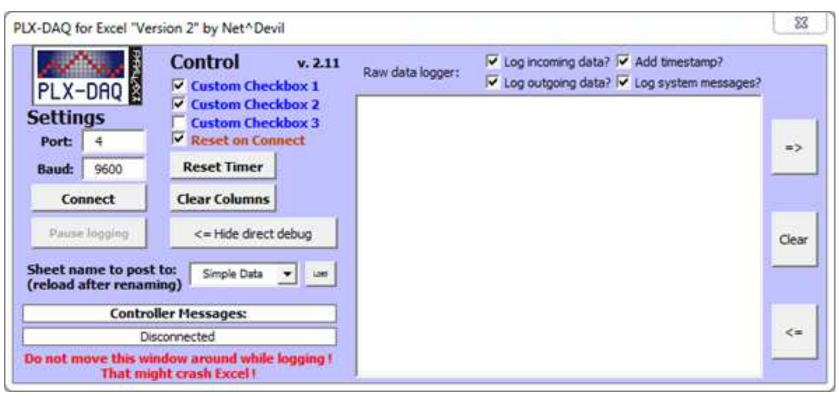

Figure 2.17. PLX DAQ control panel

The main settings and functions are:

- PORT: set to Arduino port.
- BAUD: set the baudrate running on the board.
- CONNECT: this button connects to the Arduino board and starts logging.

[13]Net Devil, *Beginners Guide to PLX DAQ v2*

- PAUSE LOGGING/RESUME LOGGING: when connected will pause the logging of data.
- CLEAR COLUMNS: will delete all logged data form the sheet. Won't clear the labels of the columns.
- RESET ON CONNECT: the checkbox should be ticked at all time. If it is ticked, the first command that Excel sends to Arduino is the reset command, thus the code restarts from the beginning. In this way you can have a fresh session. If you want to connect to your Arduino without restarting just untick the box.
- CUSTOM CHECKBOX 1/2/3: these can be used to control your Arduino during run in any way you want. There are commands to label the Checkboxes by your Arduino and to query the state of the boxes There are special commands Arduino can use to query the status of the checkboxes.

For PLX DAQ v2 to work correctly your Arduino needs to send specially formatted commends. All commands need to be sent from Arduino to the PC using the *Serial.println* commands. These commends can include parameters, variables and functions to send to as well. The commands used in the receiver code are:

- *Serial.println("CLEARSHEET")*: This command clears all data the ActiveSheet, including labels. It should be the first command on every sketch.
- *Serial.println("LABEL, label1, label2, ...")*: With this command you can set the labels for the top most row of the ActiveSheet.
- *Serial.println("DATA, DATE, TIME, data")*: This is the most basic and crucial command of PLX DAQ v2. It is used to send data from your Arduino to Excel and have it printed on the ActiveSheet. You can send anything you want but you should make sure you split the data up by commas and match the number of columns you defined with the LABEL command. "DATE" word will be switched to the current Windows computer's date, while "TIME" will be switched to the current Windows computer's time.
- *Serial.println("SAVEWORKBOOKAS,file_name")*: Will save the workbook by any given name. The new workbook will be saved in the same folder as the currently open workbook.
- *Serial.println("CUSTOMBOX1,LABEL, label_name")* and *Serial.println("CUSTOMBOX1,GET")*: You can set a label to the checkboxes on the PLX DAQ control panel, set the values to the checkboxes (ticked or not ticked) and read the value of the checkboxes (into bool).

Chapter 3

System characterization and laboratory tests

In order to check that the measurement system functions properly, respecting the requirements, it is tested under different conditions and by different operators. In this way all temperature and motion sensors are verified together with the wireless communication and solidness. A tool, Minitab v18, is used to compute the characteristics of the temperature sensors obtained from some of these tests.
Minitab was developed by Pennsylvania State University in 1972 as a teaching statics tool with a friendly graphical interface. Then it became one of the most famous tools for analyzing research data. Minitab helps companies and institutions to spot trends, solve problems and discover valuable insights in data by delivering a comprehensive and best-in-class suite of statistical analysis and process improvement tools. Combined with unparalleled ease-of-use, Minitab makes it simpler than ever to get deep insights from data[1].
All tests and experiments are performed in chemical laboratories using machines which are continuously calibrated. In this chapter the tests are explained and all results are shown.

3.1 Motion sensor tests

The three mounted tilt sensors, in charge of detecting motion of the anchor, are arranged with angles between them equal to 90° according to the xyz axis. This allows to detect the motion in all directions. A small breadboard is used for the arrangement. The figure 3.1. shows the sensors positions.

[1] https://www.minitab.com/en-us/company/

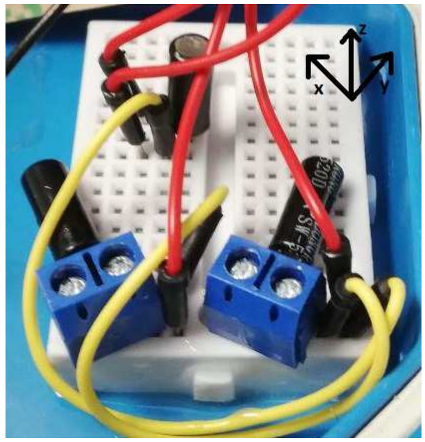

Figure 3.1. Motion senors configuration

For this test, the sender board is attached to the top of the paddles which are connected to a small motor spinner. It has different speeds from 0 rpm to 500 rpm set at the click of every 50 rpm (that is 50-100-150 rpm). The equipment used is shown in figure 3.2. In order to observe and save the data, they are transmitted, using LoRa protocol, to a receiver board directly connected to the computer, as in final code. The use of wireless communication prevents the cables of the power supply from intertwining and moreover, wireless communication is tested it in motion condition of the board during this test.

Figure 3.2. Picture of motion test

The system works with a double battery AA, connected in series to obtain a total nominal voltage of 3V, and the digital analog converter is set to compute the conversion every 5 seconds. In figure 3.2. the system is shown as working with a velocity of 50 rpm, which is the minimum speed of the test motor used. The final part of the paddle is immersed in water to simulate a condition very close to reality in the mixer, for example vibrations and motion different to rotation.
The data is collected in an excel file using *PLX DAQ v2* tool implemented in the receiver. The results for different speeds (50 and 100 rpm) are reported in figures 3.3., 3.4. and 3.5.

50 rpm	sensor_1	motion detected	sensor_2	motion detected	sensor_3	motion detected
00:00:00	242,00	0	274	1	123	1
00:00:05	301,00	1	382	1	235	1
00:00:10	269,00	1	317	1	126	1
00:00:15	217,00	1	225	1	99	1
00:00:20	285,00	1	328	1	190	1
00:00:25	294,00	0	341	1	154	1
00:00:30	214,00	1	217	1	103	1
00:00:35	288,00	1	348	1	191	1
00:00:40	297,00	0	345	0	158	1
00:00:45	217,00	1	209	1	96	1
00:00:50	274,00	1	303	1	151	1
00:00:55	268,00	0	299	0	112	1
00:01:00	221,00	1	225	1	97	1
00:01:05	236,00	1	189	1	101	1

Figure 3.3. Motion test: 50 rpm

100 rpm	sensor_1	motion detected	sensor_2	motion detected	sensor_3	motion detected
00:00:00	132,00	1	98	1	60	1
00:00:05	267,00	1	277	1	139	1
00:00:10	255,00	1	275	0	129	0
00:00:15	279,00	1	307	1	196	1
00:00:20	256,00	1	254	1	108	1
00:00:25	257,00	0	280	1	212	1
00:00:30	276,00	1	312	1	102	1
00:00:35	290,00	1	356	1	236	1
00:00:40	225,00	1	198	1	90	1
00:00:45	277,00	1	313	1	168	1
00:00:50	224,00	1	202	1	94	1
00:00:55	237,00	1	280	1	222	1
00:01:00	243,00	0	227	1	117	1
00:01:05	234,00	0	280	1	130	0

Figure 3.4. Motion test: 100 rpm

50 rpm - STOP	sensor_1	motion detected	sensor_2	motion detected	sensor_3	motion detected	
00:00:00	295	1	333	1	214	1	
00:00:05	237	1	281	1	111	1	
00:00:10	338	1	435	1	357	1	Stop button pressed
00:00:15	249	1	279	1	112	1	
00:00:20	249	0	280	0	111	0	
00:00:25	244	0	278	0	110	0	
00:00:30	247	0	278	0	114	0	
00:00:35	247	0	277	0	109	0	
00:00:40	247	0	274	0	109	0	
00:00:45	249	0	282	0	117	0	

Figure 3.5. Motion test: stop

In the last figure, the impeller has the speed equal to 50 rpm and, after 10 seconds, the stop button was pressed and the paddle stopped rotating. As it can be observed,after 10 seconds the sensors did not detect any motion, as the new value should be +/-15 greater or minor than the old value, as explained earlier in order to prevent detection of false motions or vibrations.

3.2 Static temperature tests

In this section static temperatures are measured to characterize the sensors. Static temperatures tests are tests where the temperatures of the samples do not change thus stable temperature is measured. The calibration of each sensor is done by the vendor, as reported in the specifications, thus, the offset error will not be adjusted. This can be observed in the results of the tests where the errors of the measurements are not higher than 0.5°C as from specifications.

3.2.1 Descriptive statistics

To perform these first tests, the sampling jars, which are full of demineralized water ($\sigma = 0{,}067\frac{\mu S}{cm}$), are inserted for a minimum of 8 hours in climate chambers, which error is less than 0.1°C, at temperatures equal to 40°C, 50°C, 70°C and 90°C. Before each test, the temperature of the sample is measured with a thermometer to be sure that the climate chamber works fine and the sample has the correct temperature. The accuracy of the thermometer is 0.05°C.
For every sensor, 30 measurements are collected for every single temperature (40°, 50°, 70° and 90°) for a total of 1080 collected measurements. When a sample is used for a measurement, it is reinserted in the climate chamber for 8 hours, in order

to reach the right temperature once again. The results are entered in a Minitab worksheet and then a "*descriptive statistics*" is run. The "*descriptive statistics*" is used to summarize numeric data with a variety of statistics such as the sample size, mean, median, and standard deviation. Tables 3.1., 3.2., 3.3. and 3.4. show the data collected.

Sensor	Mean	SE Mean	StDev	Variance
1	39,997	0,0176	0,104	0,0109
2	40,003	0,0171	0,101	0,0103
3	40,000	0,0174	0,103	0,0106
4	39,994	0,0174	0,103	0,0106
5	39,977	0,0164	0,097	0,0095
6	40,000	0,0205	0,121	0,0147
7	40,006	0,0164	0,0968	0,0094
8	39,997	0,0176	0,104	0,0109
9	40,000	0,0174	0,103	0,0106

Table 3.1. Descriptive statics 40°C

Sensor	Mean	SE Mean	StDev	Variance
1	49,997	0,0176	0,101	0,0103
2	50,009	0,0171	0,101	0,0102
3	50,017	0,0176	0,104	0,0109
4	50,000	0,0159	0,0939	0,0088
5	50,000	0,0159	0,0939	0,0088
6	50,003	0,0176	0,104	0,0109
7	49,991	0,0138	0,0818	0,0067
8	49,997	0,0171	0,101	0,0103
9	50,017	0,0176	0,104	0,0109

Table 3.2. Descriptive statics 50°C

Sensor	Mean	SE Mean	StDev	Variance
1	69,983	0,0167	0,093	0,0083
2	69,997	0,0162	0,0890	0,0079
3	69,982	0,0167	0,0913	0,0083
4	70,000	0,0186	0,102	0,0103
5	70,000	0,0186	0,102	0,0103
6	69,970	0,0187	0,102	0,0104
7	70,007	0,0203	0,111	0,0124
8	69,983	0,0167	0,0913	0,0083
9	69,983	0,0167	0,0913	0,0083

Table 3.3. Descriptive statics 70°C

Sensor	Mean	SE Mean	StDev	Variance
1	90,007	0,0235	0,128	0,0165
2	90,013	0,0224	0,122	0,0150
3	89,993	0,0179	0,0980	0,0096
4	89,980	0,0227	0,124	0,0154
5	89,980	0,0227	0,124	0,0154
6	89,990	0,0251	0,137	0,0189
7	89,997	0,0242	0,133	0,0176
8	90,007	0,0235	0,128	0,0165
9	89,993	0,0179	0,0980	0,0096

Table 3.4. Descriptive statics 90°C

The mean is used to describe the sample with a single value that represents the center of the data. Therefore the mean is the average of the data, which is the sum of all the observations divided by the number of observations.
The SE mean is the the standard error of the mean which estimates the variability between sample means that you would obtain if you took repeated samples from the same population. Whereas the standard error of the mean estimates the variability between samples, the standard deviation measures the variability within a single sample.
The standard deviation (stDev) is the most common measurement of dispersion, or how spread out the data is from the mean. The symbol σ is often used to represent the standard deviation of a population, while s is used to represent the standard deviation of a sample. Variation that is random or natural to a process is often referred to as noise. The standard deviation can be used to determine how spread out the data is from the mean. A higher standard deviation value indicates greater

spread in the data. A good rule of thumb for a normal distribution is that approximately 68% of the values fall within one standard deviation of the mean, 95% of the values fall within two standard deviations, and 99.7% of the values fall within three standard deviations.
The variance measures how spread out the data is from their mean. The variance is equal to the standard deviation squared σ^2.

In the previous tables, the mean can be different from the temperature of the sample at the maximum of 0.030°C and the standard deviation minor to 0.15°C, that means 99.7% confidential interval, as it is the expectation by the design and the requirements.

3.2.2 Gage R&R study

In this subsection the *gage R&R study* is performed. The *gage R&R study* helps investigate:

- *Repeatability:* how much variability in the measurement system is caused by the measurement device.
- *Reproducibility:* how much variability in the measurement system is caused by differences between operators.
- Whether your measurement system variability is small compared with the process variability.
- Whether your measurement system is capable of distinguishing between different parts.

Therefore a *gage R&R study* is used to assess the measurement system for collecting continuous data. It is used where "gages" or devices are used to measure important characteristics that are continuous. The study describes the following characteristics reported earlier. The repeatability is the variation due to the measurement device and it is the variation that is observed when the same operator measures the same part many times, using the same gage, under the same conditions. So it is the inconsistency in how a given person takes the measurement: lots of inconsistency means high variation and low repeatibility. The reproducibility is the variation due to the measurement system. It is the variation that is observed when different operators measure the same part many times, using the same gage, under the same conditions. Thus, it is the inconsistency in how different people take the measurement: lots of inconsistency means high variation and low reproducibility. The part-to-part variation is the physical or actual differences in the units being measured. The operator-part interaction is an interaction that causes people to measure different items in different ways, for example people of a particular height may have trouble measuring certain parts because of lighting or perspective.

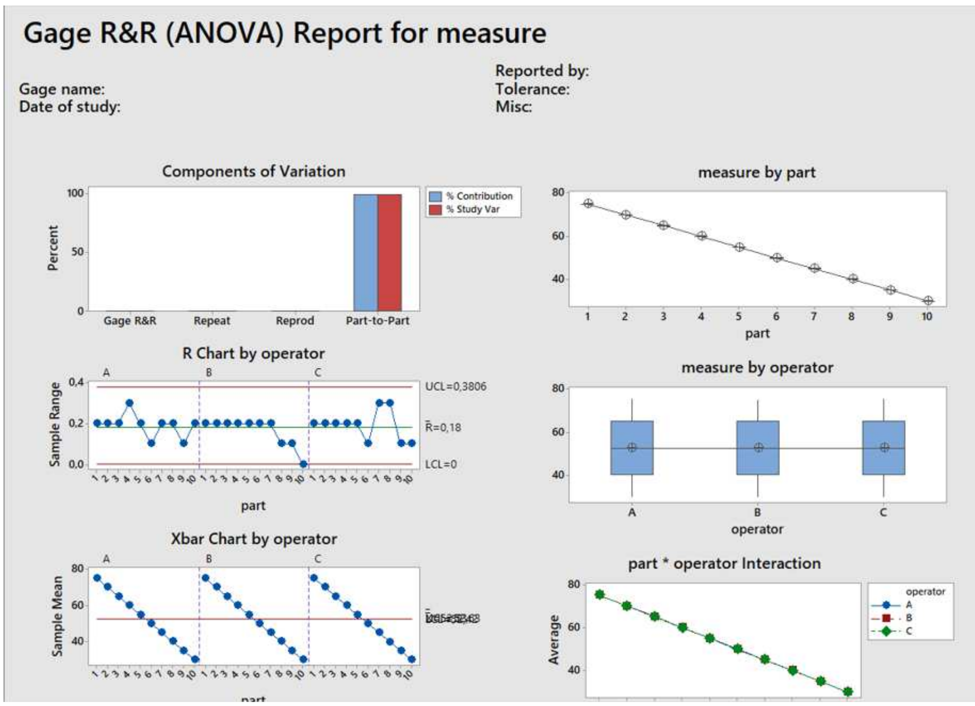

Figure 3.6. Crossed gage R&R study results

Following the steps for the descriptive statistics tests, the data is collected using climate chambers and jars fulled of demineralized water. To ensure that the data collection order does not influence the results, each operator should measure all parts randomly within a replicate. The number of operators is three. After all operators measure all parts one time, the process is repeated for all replicates. Each operator measures the samples at temperature equal to 30°C, 35°C, 40°C, 45°C, 50°C, 55°C, 60°C, 65°C, 70°C and 75°C for five times for each sensor. As before all data is entered in Minitab worksheet and the *crossed gage R&R study* is ran. The result for sensor number 1 is shown in table 3.5. and in figure 3.6.

Source	StdDev(SD)	Study Var (6 x SD)	%Study Var (%SV)
Total Gage R&R	0,0907	0,5442	0,60
- Repeatability	0,0810	0,4862	0,53
- Reproducibility	0,0407	0,2445	0,27
— operator	0,0145	0,0867	0,10
— operator*part	0,0381	0,2286	0,25
Part-to-Part	15,1553	90,9319	100,00
Total Variation	15,1556	90,93335	100,00

Table 3.5. Crossed gage R&R study results

The results of other sensors are not shown because they are very similar to sensor 1 results.
From table 3.5. and from the ones of other sensors not reported, the main weight in the total variation is the part to part source while the other sources, in percentage, are equal to 0,60%. Therefore, the repeatability and reproducibility respect the project requirements, indeed each of them are minor to the 2% on the total percentage.

3.3 Dynamic temperature tests

In this section the tests regarding the variation of temperature are performed. These tests allow to observe how sensors work when the temperature of the sample changing during a thermal cycle or immediately.

3.3.1 Difference temperature tests

In the tests, called difference temperature tests, each sensor is immersed in a sample with temperature equal to 90°C. After the sensor reaches the temperature, it is immersed in a sample with a water at room temperature 27°C. This test is repeated for all the sensors.

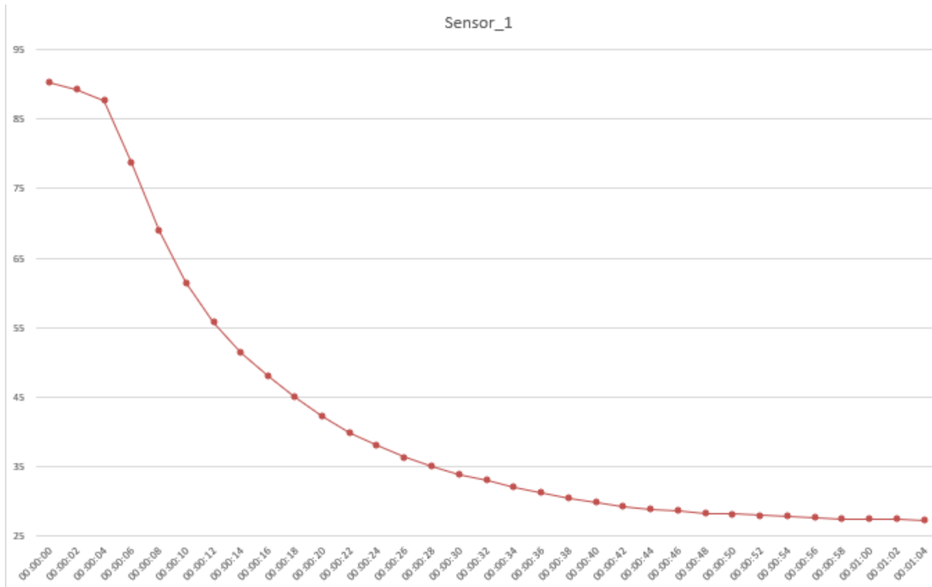

Figure 3.7. Temperature difference 90°-27°C results sensor 1

As shown in figure 3.7. the total time is equal to 1 minute and 4 seconds for a difference of around 63°C, while the time interval in the 90-10% is equal to 27 seconds. The average time of all the sensors is around 26,8 seconds.
The same test is performed in opposite way using the samples with the temperatures equal to 27°C and 85°C.

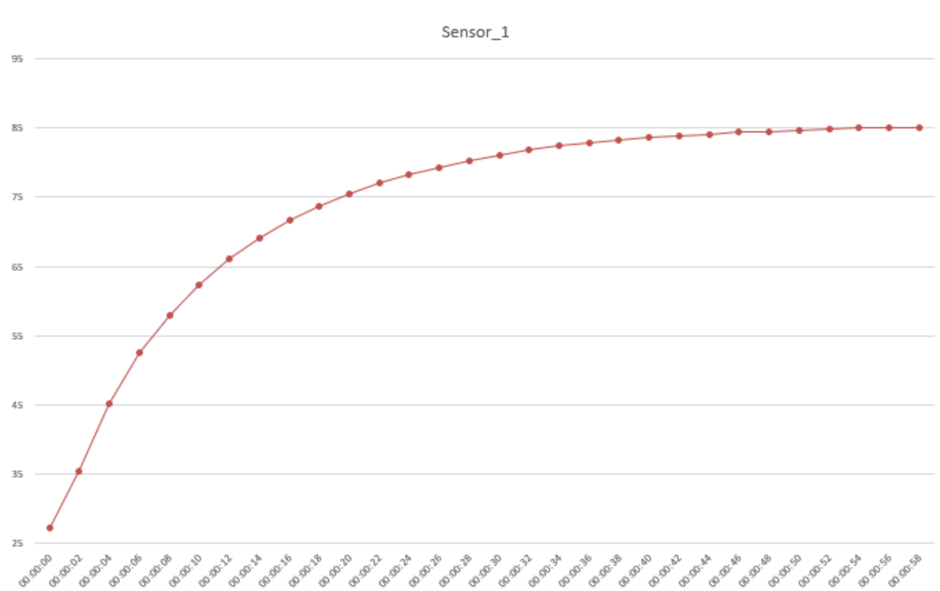

Figure 3.8. Temperature difference 27°-85°C results sensor 1

Similarly to the previous case, the total time is equal to 58 seconds for a difference of around 58° degrees, while the time interval in the 10-90% is equal to 26 seconds. The average of all sensors is around 26,1 seconds.
Just as in the previous case, the results only for the first sensor are shown in figure 3.8. since the others are very similar.
The tests show the speed of the sensors necessary for reaching and measuring the new temperature. The interval time in the 10-90% of range is always minor to 30 seconds and in 2 seconds the sensor temperature can decrease or increase of 10°C. Therefore, the sensors are fast enough to work with a sampling frequency equal to 30 mHz, without losing any relevant temperature gradients.

3.3.2 Thermal cycle test

In order to test the correct functionality of the entire system, similar industrial thermal cycles are reproduced. To increase the temperature of the sampling water ($800ml$), a heating slab under the beaker, which contains the liquid, is used. The heating system is controlled by a thermometer probe connected directly to the slab. The temperature probe has a display to set and observe the temperature. The sensor being tested is hold, using a plastic tie, on to the thermometer probe. The maximum difference between the temperature probe and the sensor, during the thermal cycles, is about 0.3°C, and it is acceptable since they both have an accuracy equal to 0.5°C. Figure 3.9. shows the material used.

The slab is set to 350°C while the temperature setpoint is equal to85°C. When the sensor reaches the temperature setpoint, the heating system is turned off. This test is performed to see not only the behavior of each sensor in a thermal cycle but also to test how strong they are.
For each sensor the test is repeated 5 times. Therefore the initial robustness is verified and the sensors can be mounted together to start the tests in the pilot mixer. Figure 3.10. reports the temperatures collected by a sensor in a thermal cycle.

Figure 3.9. Material for thermal cycle test

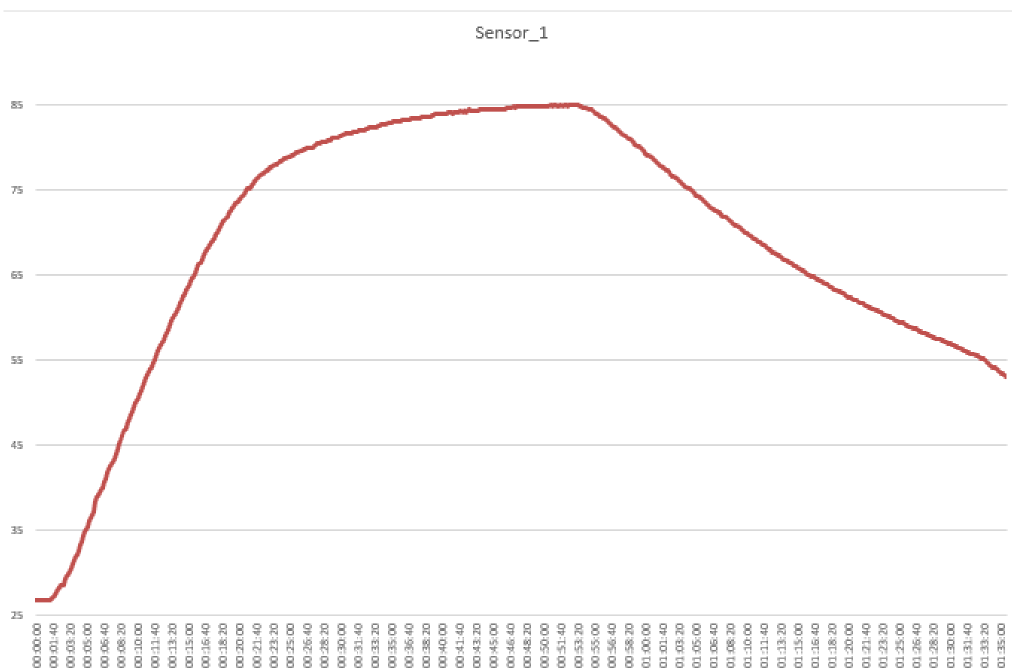

Figure 3.10. Temperature thermal cycle test

Chapter 4

Tests in pilot mixer

In this chapter experiments and tests of the designed system are performed using the pilot mixer, which is a small reproduction of industrial mixers used nowadays with some differences. The purpose of these experiments is not only to test the sensors but also to collect data for future fluid dynamic simulations to improve the mixing process in terms of cost and quality.
Before proceeding with these tests, an explanation about the pilot mixer structure and how the system is implemented and mounted in the pilot is given. The solution adopted in this chapter is not the final one because it does not respect all GMP requirements. A possible GMP solution is described in the future steps in the next chapter.

4.1 Pilot mixer

The pilot mixer used for the following tests is shown in figure 4.1., while figure 4.2. shows its technical drawing. The internal dimensions of the vessel are 40 cm for its diameter and 58 cm for its height, so the maximum capacity is around 80 kg. This pilot mixer has a single rotating anchor with three arms and the maximum impeller speed is 28 rpm. At the end of the anchor, a marine-propeller-shaped propeller is installed and it rotates in the opposite direction with respect to the anchor, with a maximum speed of 997 rpm. In addition, this mixer has a pump for liquid recirculation. It pumps the fluid from the bottom to the top of the mixer. The speed of these systems is set by a graduated potentiometer mounted on its control panel.

Figure 4.1. Pilot mixer used

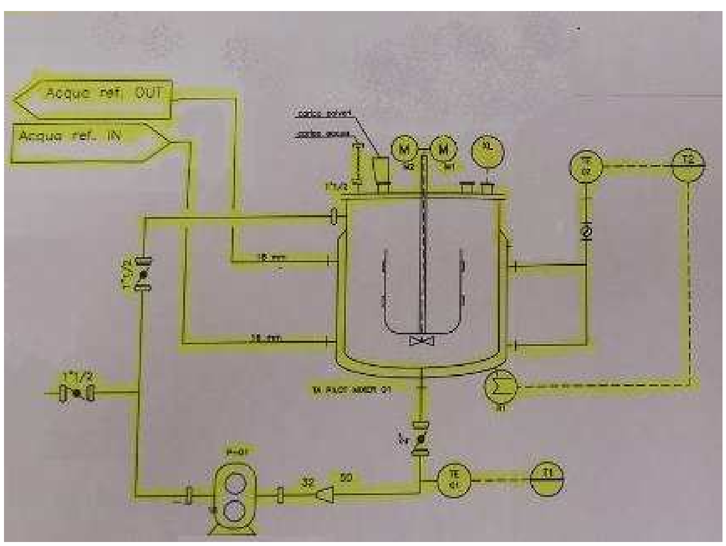

Figure 4.2. Pilot mixer drawing

The heating system of pilot mixer is different from the industrial one, indeed, to increase the temperature, a thermal oil in the jacket is heated by the usage of a resistance positioned at the bottom of the vessel. Vice versa, to decrease the temperature, cold water from the chiller flows into the half-pipes system in the jacket and in the end it returns back to the chiller. The chiller temperature is set to 5°C. Figure 4.3. shows the electric panel which controls the pilot mixer.
The pilot mixer has two temperature sensors, one to measure the oil temperature in the jacket, and one for the product. The latter one is able to measure the temperature only if the manual valve at the bottom of the mixer is open. This means that the recirculation system should be activated in order to have a flow of the product inside the pipe where the sensor is connected. Figure 4.4. shows the position of the sensor.

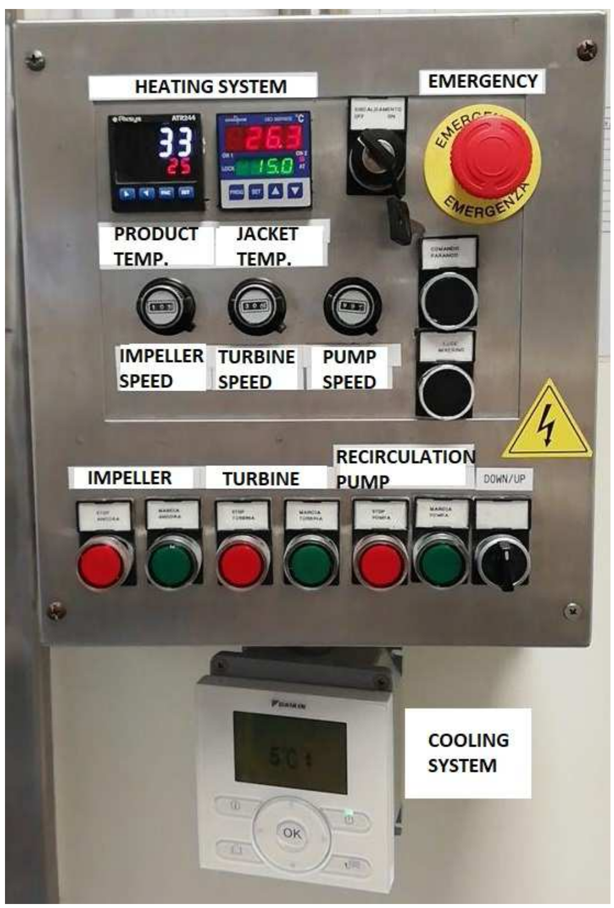

Figure 4.3. Pilot mixer panel

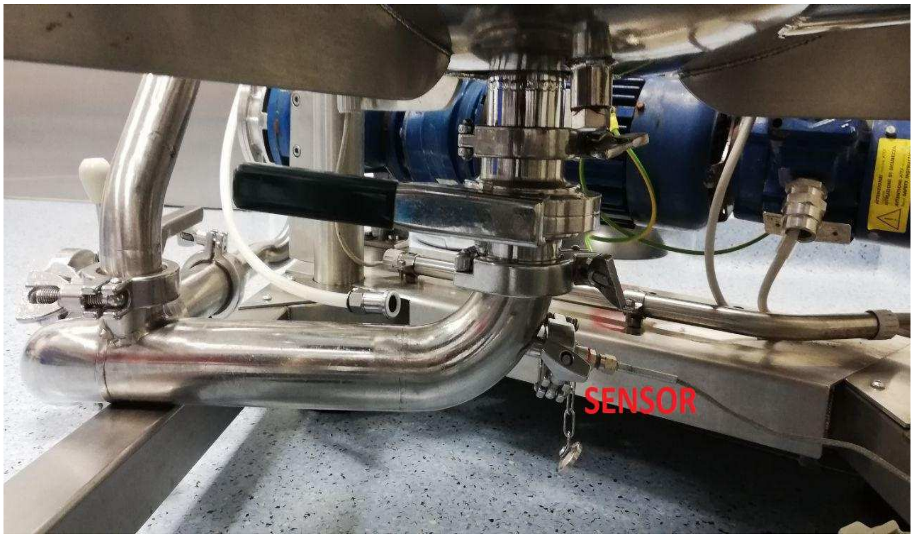

Figure 4.4. Temperature sensor piping

4.2 System integration

In order to obtain maximum flexibility to decide the position of the sensors in width and height, the sensors cables are connected in a parallel way at the top of the mixer. This leads to difficulty in the cleaning process after the tests but more flexibility to decide in which points the temperature should be measured. Thus the cleaning phase will be performed manually (possible in the pilot mixer).
The sensors cables are long enough to measure the temperature in all points of the anchor, so, since they are waterproof and covered with PTFE material, resistant up to 150°C, no changes are made in this way.
The number of sensors during this test phase is seven due to the small dimension of the tank, indeed two sensors are positioned on each arm of the anchor and one at the bottom of the anchor as reported in figure 4.5.

The Arduino boards have an operating temperature range of −10°C to 80°C, but the temperature in the system can reach an even higher temperature until 100°C, and they are not chemical resistant, thus a thermal and chemical protection is needed.

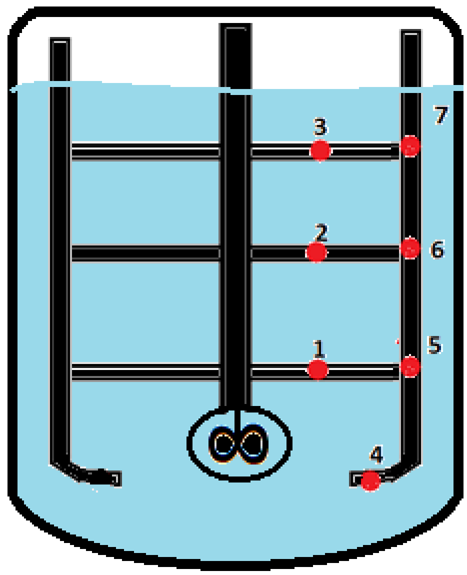

Figure 4.5. Sensors positions

4.2.1 Temperature and chemical protection

In military and aerospace application a special coat is used to protect the electronic hardware. It is called conformal coating which is a thin polymeric film applied to a printed circuit board in order to protect it. The film is typically applied at $25 - 250\mu m$ and conforms to the shape of the board and its components, covering and protecting solder joints, the leads of electronic components, exposed traces and other metallic areas from corrosion.
Different materials can be used but polyurethane is the best one for its excellent moisture and chemical resistance and good dielectric properties and abrasion resistance. The conformal coating applications are several:

- Manual spraying: conformal coating can be applied by an aerosol can or hand-held spray gun. This method is operator dependent so it is not so precise;
- Automated spraying: programmed spray system that moves the board on a conveyor under a reciprocating spray head that applies a conformal coating;
- Selective coating: an automated conformal coating process that uses programmable robotic spray nozzles to apply the conformal coating to very specific areas on the circuit board;

- Dipping: the circuit board is immersed then withdrawn from the conformal coating solution. This method is only practical when coating on both sides of the board is acceptable;
- Brushing: it is a simple application technique used mainly in repair and rework applications. The conformal coating is applied with a brush to specific areas on the board[1].

Therefore the conformal coating protects boards from the environment and temperature and also reduces its deterioration caused by the external factors in its operating environment.

The conformal coating chosen is *RS 494-714* by *RS Components* that is a flexible and transparent modified silicone conformal coating spray. It has been formulated to work in high temperature ambient and it is resistant to many solvents used in industries. In addition it can be reworked.
According to the component datasheet: the operating temperature range is from -70°C to 200°C, the dissipation factor (@ 1 MHz, 25°C) is 0.039 and dielectric constant equal to 4. The low dielectric constant allows the use of the conformal coating also on the antenna.

Unfortunately, the polyurethane coating does not respect the GMP requirements, because it can compromise the product. Therefore, this protection method can be used only for a prototype or, in the industrial mixers, only if it is not in contact with the product.
The board mounted on the top of the anchor can be encapsulated in a custom box made of material which respects the GMP requirements. A specialized company (*3M*) is able to print in 3D using PTFE, so the shape of the box can be studied according to the dimensions of the anchor avoiding product depositions and respecting the requirements of the automated cleaning and sanitization phases of the mixer.
Another material, which can be used to manufacture the box, is the AISI 316L, but in comparison to the previous one, it is a conductor, so the antenna cannot be incapsulated inside the box, while in polytetrafluoethylene box can.

For time and cost reasons, the production of the printed PTFE box is included in future steps, while for the test a plastic ATEX box is used to encapsulate the board, including the antenna, with the battery packs and the motion sensors.
The ATEX directive consists of two EU directives describing what equipment and work space is allowed in an environment with an explosive atmosphere. ATEX derives its name from the French title of the 94/9/EC directive: Appareils destinés à

[1] https://www.techspray.com/the-essential-guide-to-conformal-coating

être utilisés en ATmosphères EXplosives[2].
The box is IP67 that means it is dust tight (IP6x) and the ingress of water in harmful quantity shall not be possible when the enclosure is immersed in water under defined conditions of pressure and time (up to 1 m of submersion)[3](IPx7). The box with its connections is shown in figure 4.6.

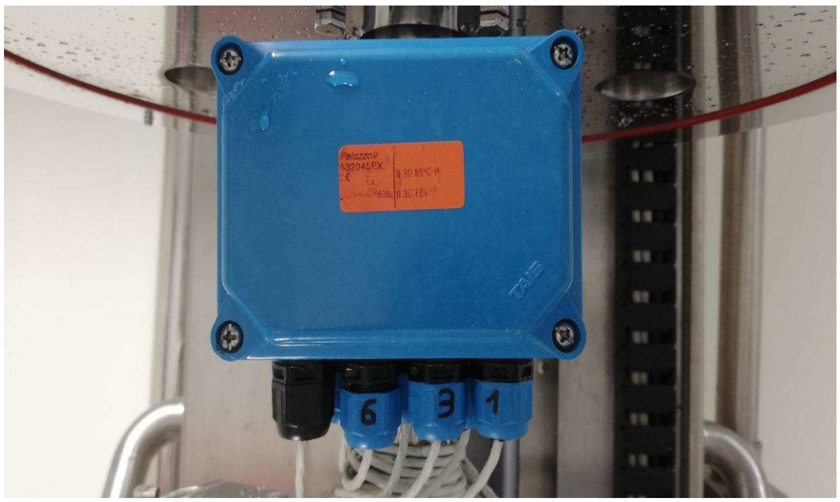

Figure 4.6. ATEX box

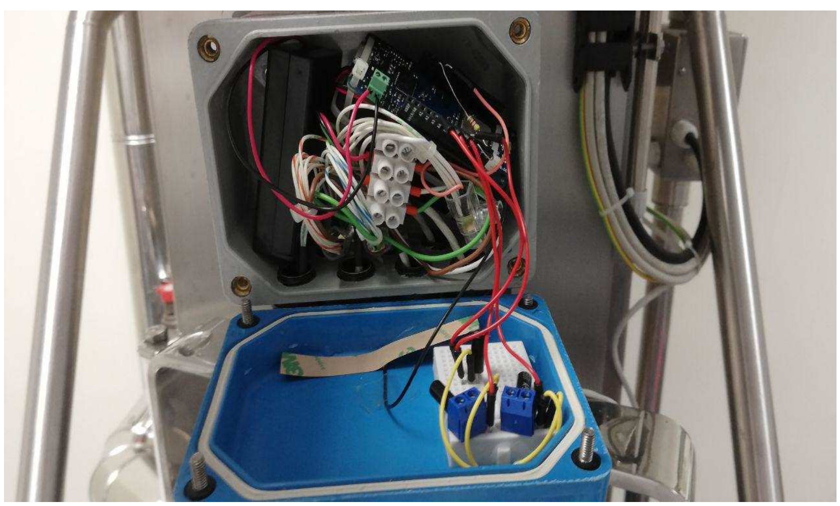

Figure 4.7. Inside the box

[2]Directive 2014/34/EU of the European Parliament and of the Council of 26 February 2014 on the harmonisation of the laws of the Member States relating to equipment and protective systems intended for use in potentially explosive atmospheres (recast) (OJEU L 96, 29.3.2014, p. 309).

[3]IEC 60529 standards from International electrotechical commission

In order to bring the sensors outside the protection box, seven holes have been made on it and to keep the IP67 standard of the box, watertight cable glands are used, as shown in figure 4.6. To verify the waterproofness, the system was immersed in water for 15 minutes and no water trace was found inside the box.
Internally, on the cover of the box, the motion sensors is installed and the two battery packs are included, one connected to the sensors and one to the board. Then all connections are performed. Figure 4.7. shows the components and the wiring inside to the box.

Figure 4.8. Test configuration

Thanks to a metal clamp soldered at the back, the box is held on to the top of the anchor, while the sensors are positioned using the plastic ties. Figure 4.8. shows the complete configuration for the tests.
The receiver board, in this case, is positioned outside the mixer with the antenna above the glass screen on the top of the mixer. This screen is usually used to observe the inside of the mixer during the blending process.

4.3 Tests with water

The first tests are performed with demineralized water which has viscosity equal to $1cp$ at 20°C. The mixer was filled with water (80 Kg), so all sensors are immersed in fluid. Sensor 7, which is the highest sensor, has 4 cm of water above it. Then a thermal cycle is performed and the data, from the sensors, is acquired and saved. The heating system is set to 85°C, while, to decrease the temperature, the water enters in the half-pipe system at 5°C from the chiller.
The first test is performed with all the motion systems (anchor, propeller and water recirculation) at a stop. Figure 4.9. shows the data collected by the sensors during heating until the average temperature of the water exceeds 85°. This test is performed in order to observe the possibility of heating the water at the beginning of the process, without any other materials and without using energy to fuel the motion system (anchor, propeller).

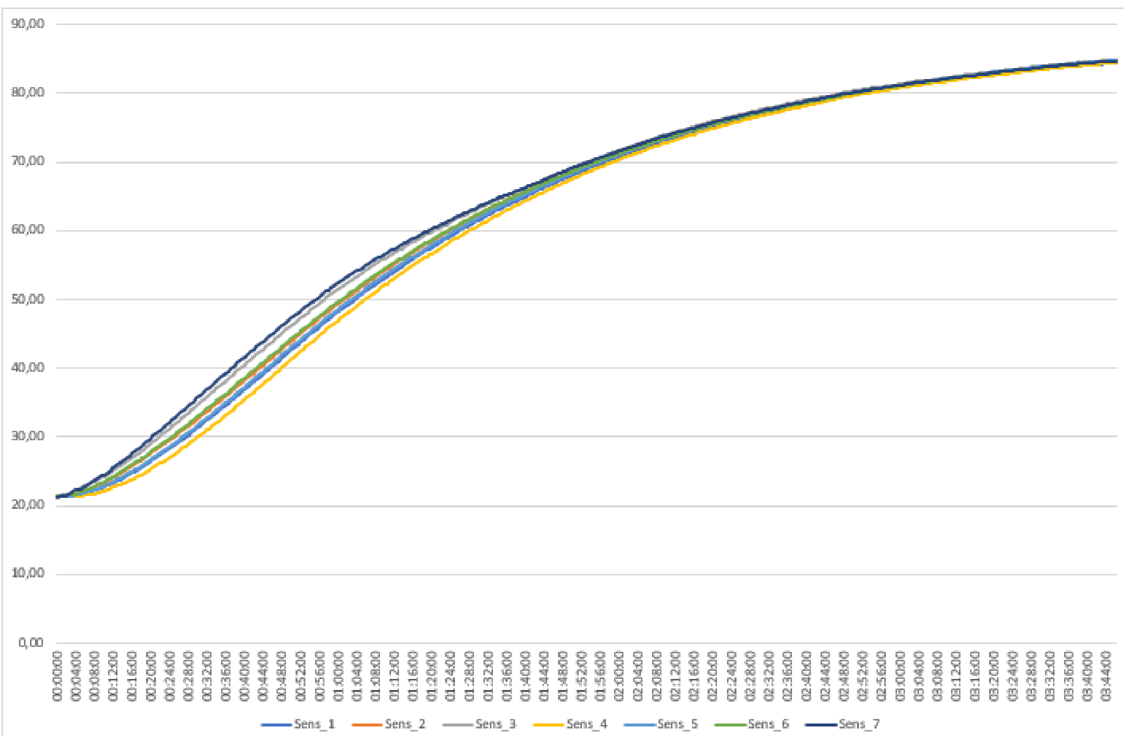

Figure 4.9. Water with no motion (heating)

As expected, the temperature gradients between the top sensors and the bottom can be observed. At time 0 the temperature difference between sensor 7 and 4 is null, during the process the difference increases up to 6°C when the average temperature is around 45°C, then it decreases and at the end it is below the accuracy of the sensor. This phenomenon can be explained by the convection and the dissipation of temperature. In fact, up to a temperature of 45°C, the convection is the most important heat transfer phenomenon in the mixer, leading to a higher hot flow to the top, decreasing the temperature at the bottom. In the second phase, heat dissipation between water and air, in the top part of the mixer, is so influential so as to reduce the speed of the increasing temperature, while the water at the bottom increases its slope, recovering the temperature gap. As regards to the gradients of the temperature between the sensors at the same level, they are not visible because the short distance does not allow them to be detected or observed. The sensors on the same arm of the anchor have a distance around 20 cm.

Figure 4.10. reports the data collected in the cooling phase.

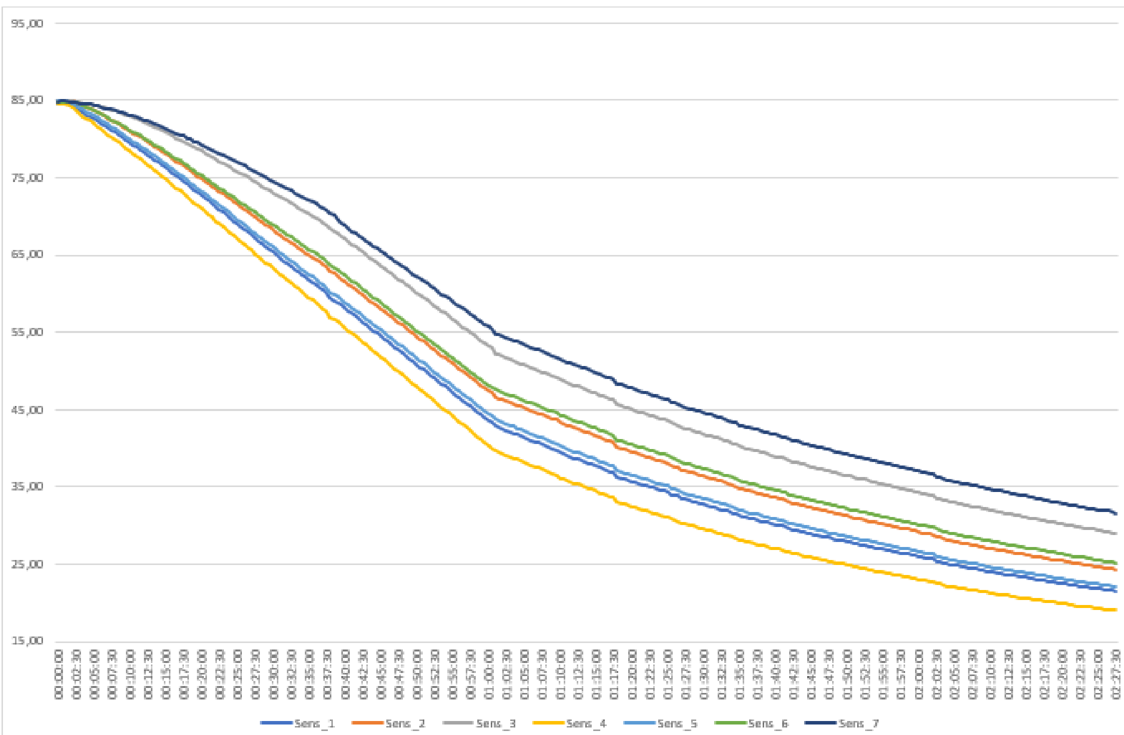

Figure 4.10. Water with no motion (cooling)

The temperature difference between the sensor at the beginning is equal to zero, but it immediately decreases when the water at 5°C flows in the half-pipe system. The water entering at the bottom of the mixer and coming out from the top leads to a fast decrease of temperature at the bottom. This fact explains the high difference of the temperature measured by the sensors at the top and at the bottom. In this case it is not possible to expect a homogenization of the temperature inside

the mixer due to the low temperature used for the cooling. In the end the average temperature is around 24.5°C with a minimum equal to 19°, measured by sensor 4, and a maximum temperature equal to 31.5°. Therefore, without the motion of the fluid, the cooling phase takes a long in time and can arise issue connected to the temperature, in fact when the temperature at the bottom is equal to 30°C, the average temperature is around 37°, therefore the fragrance added at this point can be altered by the temperature that has an average greater than 35°C and at the top it is equal to 45°C.
This is not the worst case because, though the fluid in the mixer is always is motion, the viscosity of the demineralized water is much lower than the viscosity of one product when the fragrance is added (the fragrance is the last raw material added in the health care production).

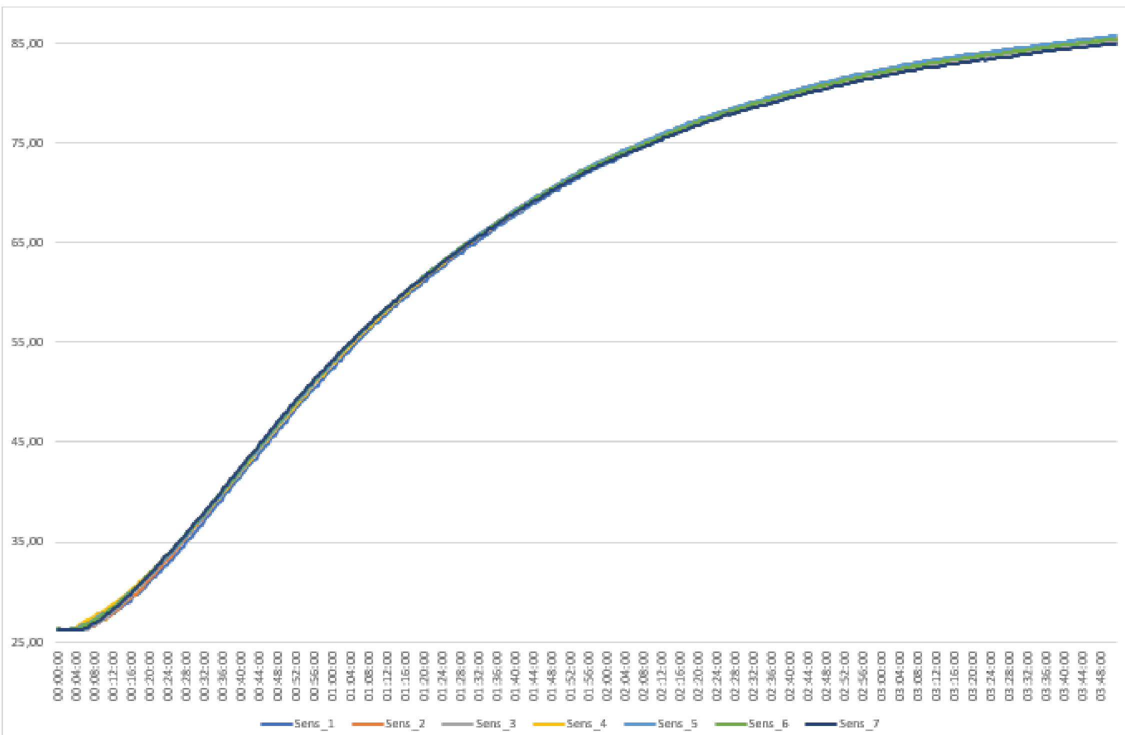

Figure 4.11. Water with anchor speed of 2,5 rpm (heating)

After the first two tests without any motion of the fluid, the tests with different speeds of anchor are performed. With the same quantity of demineralized water, and the same temperatures settings for the heating and cooling, a thermal cycle is performed with the speed of anchor equal to 2,5 rpm (the propeller and the water recycle is stopped). The result for the heating is shown in figure 4.11.

In this case, no differences of temperatures are detected, either in width or in height. This means that a low speed flow is sufficient to blending the water overlooking the convection phenomenon. There is only a difference with a maximum of around 1.2°C between sensor 4 and sensor 7. The first sensor, in this case, measures a higher temperature from 70°C to 85°. The reason for this being the heat dispersion between the water and the air inside the top of the mixer, as explained earlier.

The temperatures collected during the cooling down phase are shown in figure 4.12. During this phase, difference between the temperature at the top and at the bottom is visible especially at the beginning with a maximum equal to 4°C between the sensor 7 and 4. This difference decreases during the phase because the difference of temperature between the jacket and the fluid at the bottom is less than at the top so the temperature in the bottom of vessel decrease slowly.

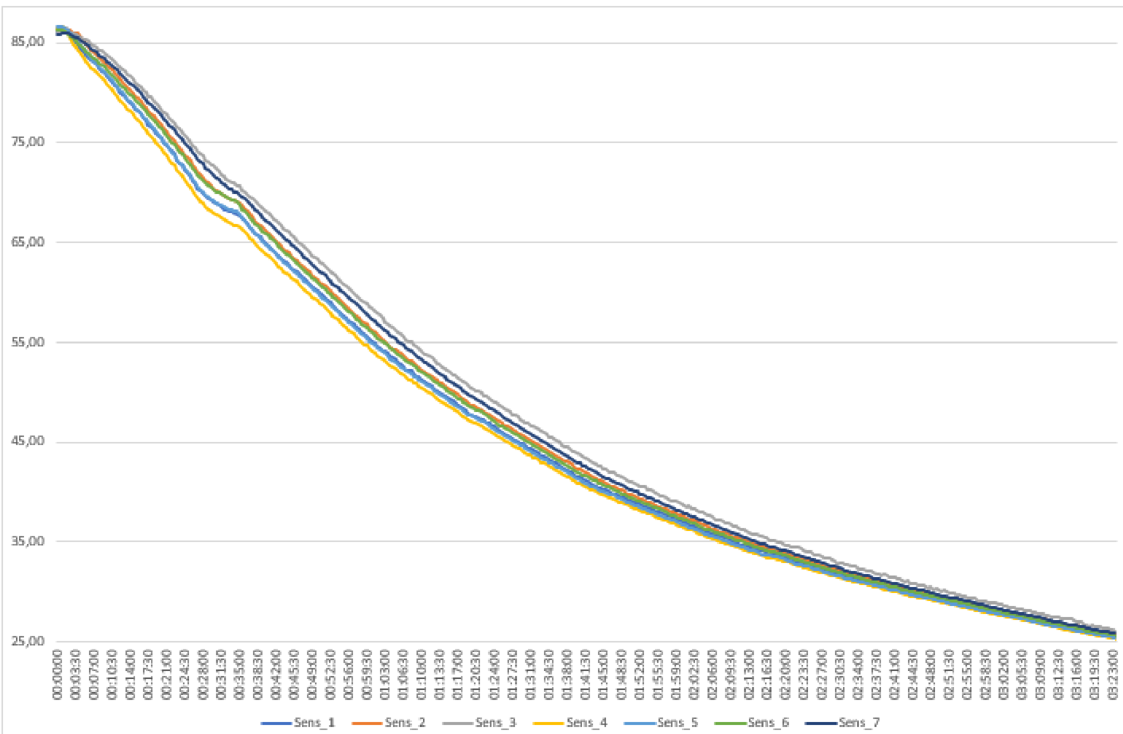

Figure 4.12. Water with anchor speed of 2,5 rpm (cooling)

To obtain a homogeneous temperature during the cooling, the speed of anchor is set equal to 15 rpm. This means that the flow is turbulent.

Heat transfer during the mixing process is especially due to convection. Convective heat transfer takes place both by diffusion and by advection. In the mixing process, the anchor and the propeller force a speed and motion of the fluid, thus, in this case, the convection is called forced. The flow in a fluid can be laminar or turbulent. In the first kind, the fluid proceeds in regular and tidy way, so there is no blend between the difference parts of the fluid in motion. Differently, in the case of turbulent flow, the trajectories of the fluid are complex and windy with continual blend processes according to the current between the fluid masses in the different zones.

Figure 4.13. shows the temperatures collected by the sensors when the temperature decreases from 85°C to 30°C with the anchor speed of 15 rpm.

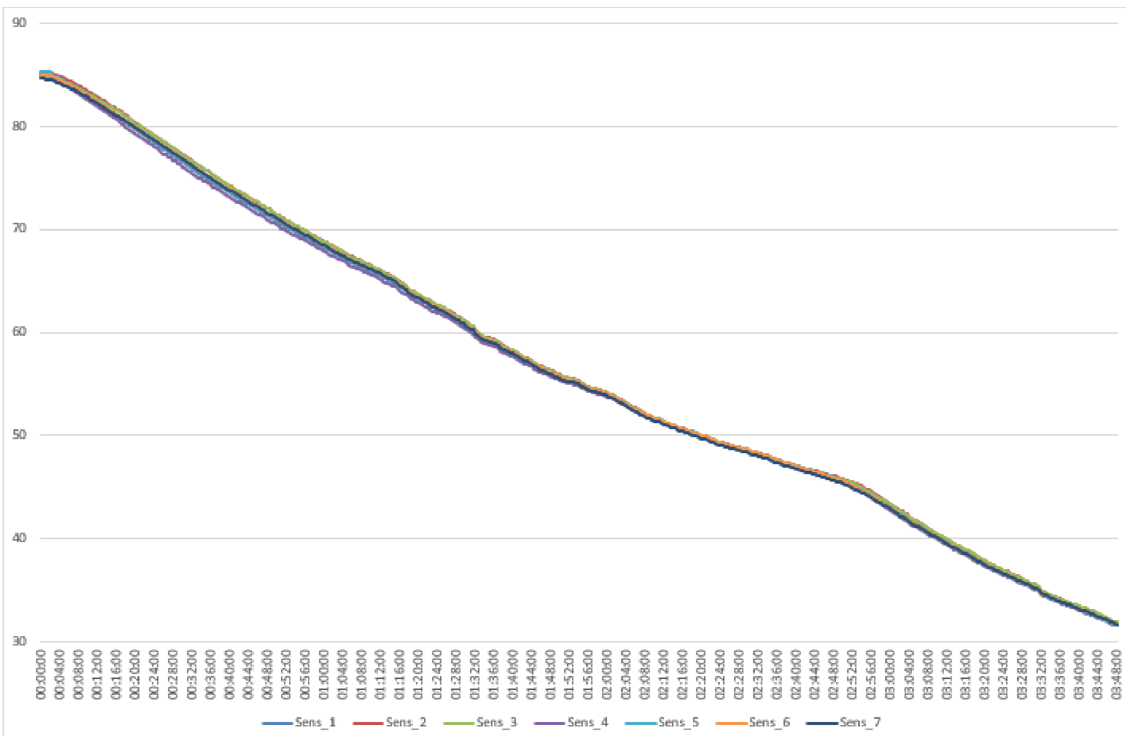

Figure 4.13. Water with anchor speed of 15 rpm (cooling)

In this case, due to the turbulent flow, the temperature in the mixer is homogeneous, thus allowing the temperature of the fluid to be measured in one point because it is the same in the whole of the tank.
After the test with only the motion of the anchor, the thermal cycle is observed with the motion of the propeller. Its speed is set to 100 rpm, with the anchor stopped. The figure 4.14. shows the heating phase.

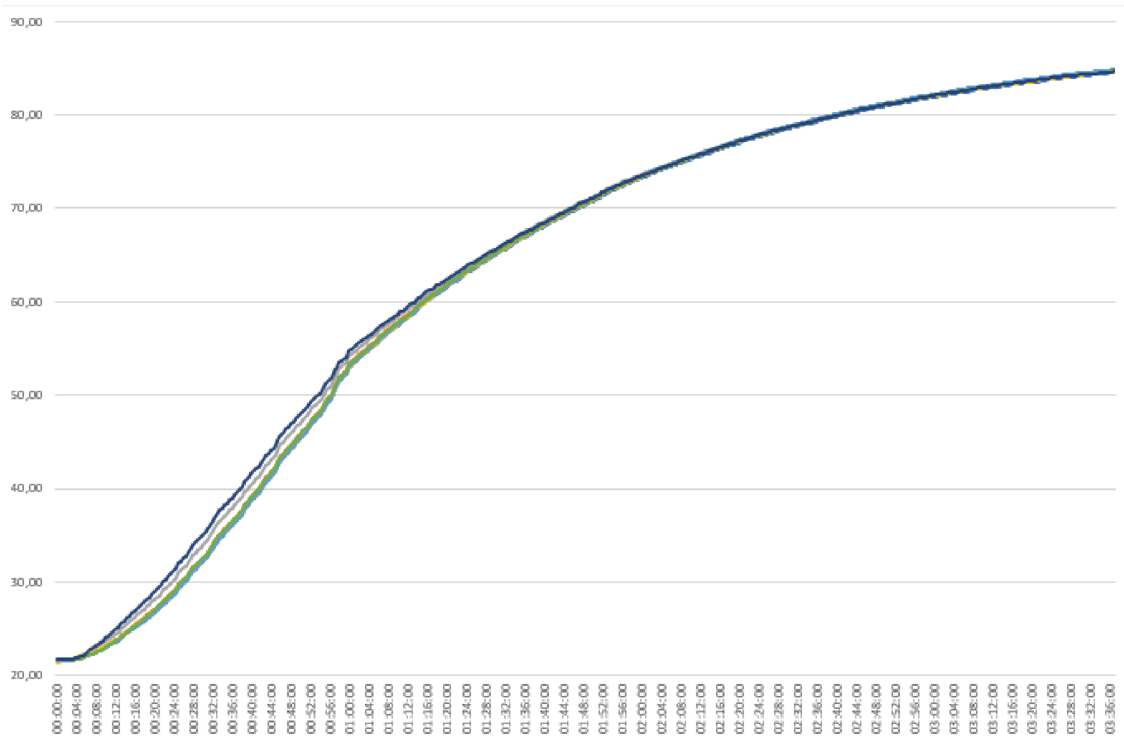

Figure 4.14. Water with propeller speed of 100 rpm (heating)

The temperature at the beginning is equal to 27.5°C without difference inside the mixer. When the heating phase starts the difference between the sensor 7 and 4 increases until reach 3°C and then decreases. The reasons are the same as for the previous cases.

In figure 4.15. the cooling phase is shown. Due to the big difference of temperature between the top and the bottom, a turbulent enough flow is not reached. Also the temperature difference between sensor 7 and 3, which is the sensor on the top arm, is visible. The particular flow created by the propeller leads to increase the difference during the cooling phase. This difference is maximum, that means it is equal to 3.5°C, when the average temperature is around 45°C.

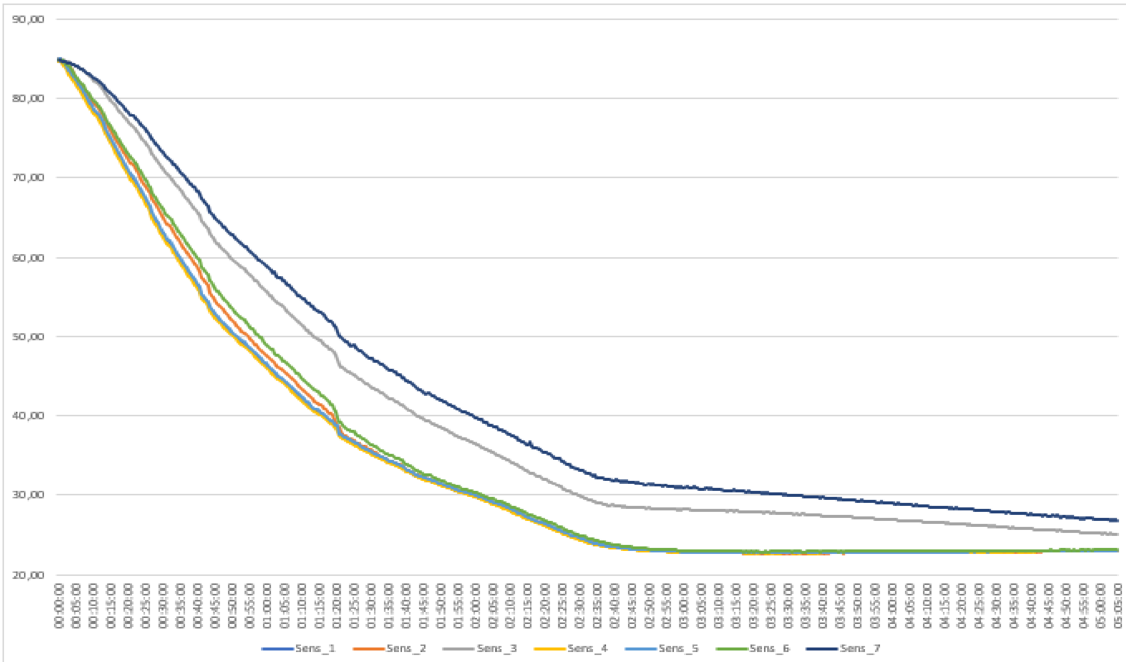

Figure 4.15. Water with propeller speed of 100 rpm (cooling)

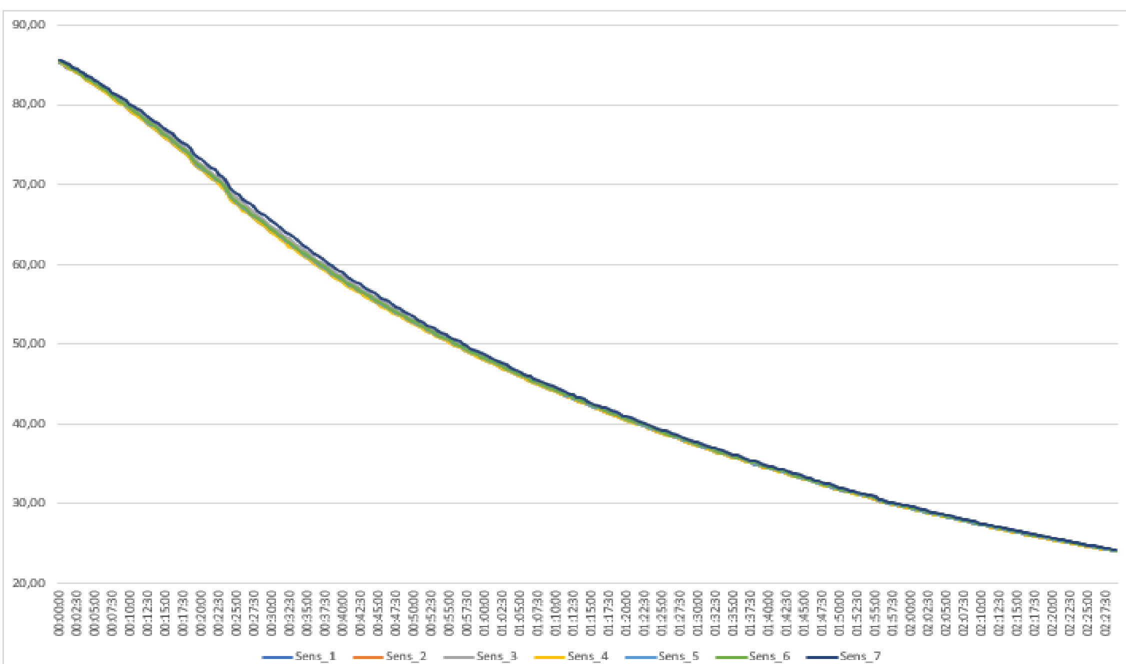

Figure 4.16. Water with propeller speed of 320 rpm and anchor speed of 2,5 rpm (cooling)

To reach a strong enough turbulent flow in the mixer with the water, the speed of propeller is set to 320 rpm and the anchor one to 2,5 rpm. The cooling phase is shown in figure 4.16. In this case a strong enough turbulent flow is reached therefore no temperature difference is visible with this system.

Tables 4.1 and 4.2 compare the time spent to heat up the water and to cool it down. The average temperature range for the heating is 30°C to 85°C, while for the cooling is 85°C and 30°C.

WATER HEATING(30°C - 85°C)	**Time**
No motion	03:22:00
Anchor 2,5 rpm	03:20:00
Anchor 15 rpm	03:15:00
Propeller 100 rpm	03:17:00
Propeller 320 rpm and anchor 2,5 rpm	03:14:00

Table 4.1. Time water heating

WATER COOLING(85°C - 30°C)	**Time**
No motion	01:56:00*
Anchor 2,5 rpm	02:42:00
Anchor 15 rpm	02:24:00
Propeller 100 rpm	02:13:00
Propeller 320 rpm and anchor 2,5 rpm	01:58:00

Table 4.2. Time water cooling (*The temperature is not homogeneous)

The times in the heating phase are very similar, in fact from no motion to the propeller and anchor case, the gain is equal to 8 minutes. This result is valid for a pilot mixer where the heating system is made up of a jacket filled with thermal oil heated by a resistance and not by a half-pipe structure with vapour at 135°C. However, for bigger dimensions and with different viscosity of the fluid in industrial mixers, a higher gain is expected.

The times in the cooling phase are very different, in fact on increasing the speed of anchor and propeller the time decreases. The time of first case is not representative because the average temperature is 30°C but it is not homogeneous, indeed sensor 7 measures a temperature around 38°C, while sensor 3 measures a temperature around 23.5°C. In the case of cooling phase the flow has an important role in the time, indeed with a very turbulent flow the time decreases, in comparison to the laminar flow case, of around 40 minutes.

4.4 Tests with cleanser

To test the system in a real production condition, a cleanser batch is performed in the pilot mixer. The thermal cycle is reported in figure 4.17.

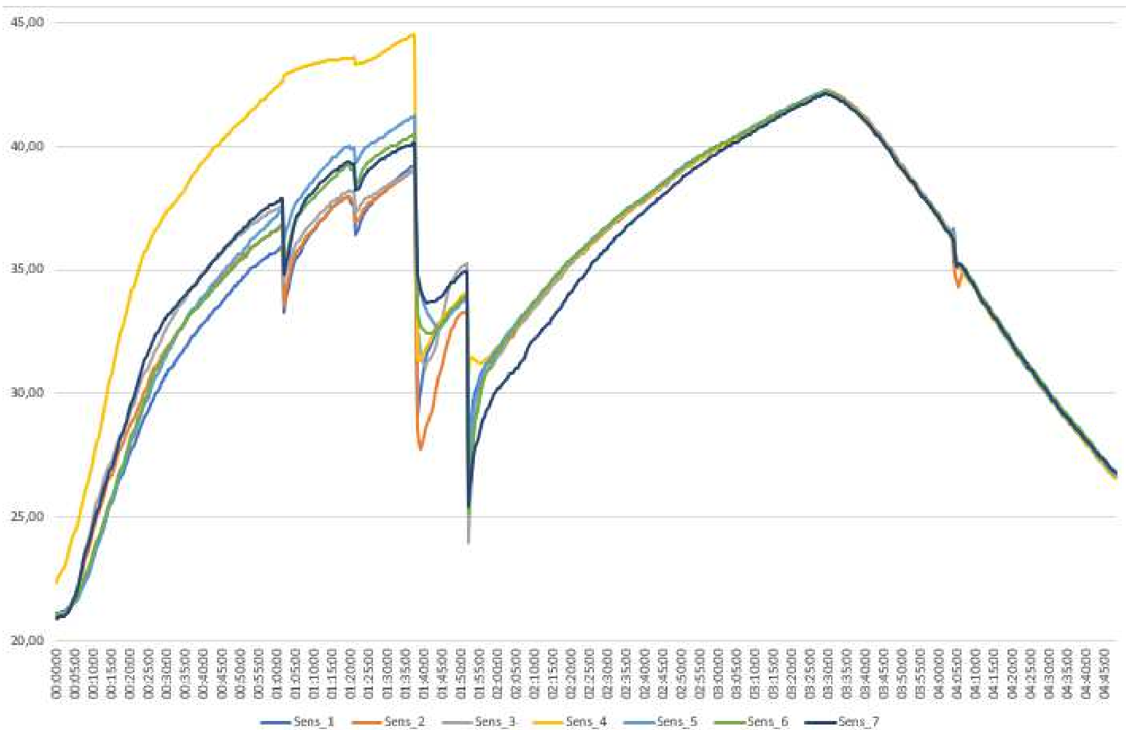

Figure 4.17. Cleanser production test

The steps to produce the cleanser are very straightforward. First of all, the raw materials are weighed to produce 80 kg of product, in order to fill up the mixer, then around 16 kg of demineralized water is heated up between 40°C and 45°C the jacket is set to 45°C to maintain the temperature of the liquid high enough. The speed of the anchor is set to 6 rpm. With this quantity of water only sensor 4 is immersed in water and, therefore, it is the only one measuring the temperature of the fluid. The other sensors measure the temperature of the air contained in the mixer.
When the water reaches the right temperature, at 01:02:00, a powdered raw materials (320 g) is inserted in the mixer, by opening the glass window on the cover. For this reason, the temperatures measured by sensors in the air decrease, as the hot air is dispersed outside the machine. After adding the powdered raw materials, the propeller is set to 300 rpm while the anchor to 28 rpm. The only sensor immersed in the fluid is always sensor 4.

When the raw material is dissolved in water, at 01:21:00, another powdered raw material (400 g) is added in the same way as in the previous case. When all substances are melted, at 01:38;00, two liquid raw materials (5 kg and 27 kg) are added and also sensor 3 and 5 are immersed in the blending fluid. In this step the propeller is stopped to avoid the creation of a lather. At the 01:52:00 all the remaining raw materials (41 kg of water and 4 kg of other materials) are added in the same way as in the other cases. From now on, all sensors are immersed in the fluid. After the complete blending of the product, the cooling phase starts at 03:37:00. When the product reaches the temperature of 36°C, at 04:03:00, the ph and viscosity is adjusted with the chemical materials. In this case fragrance is not added.

From figure 4.17. the variations of temperature can be observed. When the window on the top of the mixer is open, the temperature measured by the sensors in the air decreases because the hot air flows outside the mixer. All raw materials have a temperature equal to 25°C when they are added leading to a decrease in temperature of the total fluid.
Another phenomenon which can be observed in the graph is the different speeds to increase the temperature according to the fluid. The slope to heat the water is steeper than the slope when all materials are added, although the entire jacket at 01:52:00 has a temperature equal to 45°C and the speed of the anchor is higher. The reason is attributed to the different physical characteristics of the fluid, in fact the viscosity at the end is higher and the blending fluid is not homogeneous.

After the realization of the cleanser, a thermal cycle is performed with it, in order to observe how the viscosity influences the temperature gradients inside the mixer. The viscosity of the cleanser is around 3500 cps. The figure 4.18. shows the data collected during the heating phase with a speed of anchor equal to 2,5 rpm and the temperature setpoint set to 85°C.

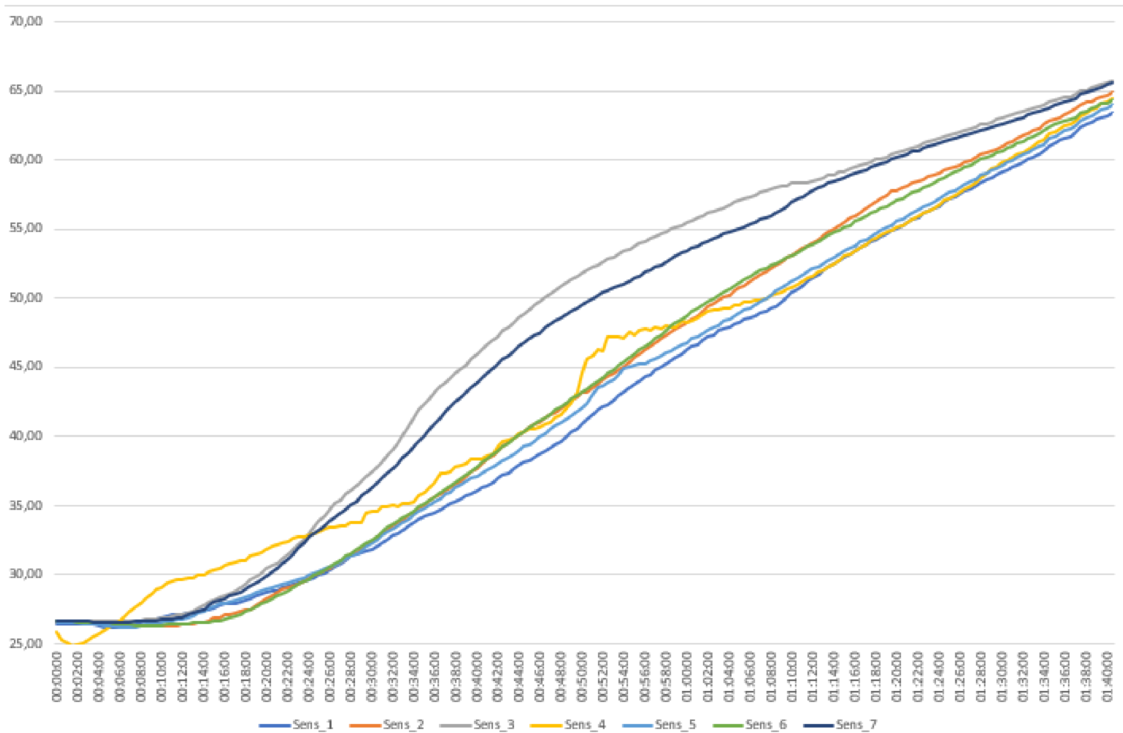

Figure 4.18. Cleanser with anchor speed of 2,5 rpm (heating)

At the beginning all the sensors measure the temperatures at around 26.5°C. When the heating starts, the sensor 4 at the bottom of the anchor, increases its temperature faster than the others which in the first 5 minutes keep the same temperature. This phenomenon is caused by the thermoresistant position, since it is at the bottom, in fact the temperature of the oil at the bottom of the jacket is higher than at the top and, moreover, due to viscosity, convection is not present. Furthermore, due to the high gradients between the fluid at the top and at the bottom, convection starts and the sensors on the top increase the temperature faster than the others. After the 60°C, due to the heat lost by the top fluid with the air, the temperature of the fluid starts homogenizing, just like in the cases with water.
The temperature measured by sensor 4 increases and decreases during the phase, a possible explanation could be linked to the low speed of the anchor that is not able to rotate and to blend the fluid mass in the bottom of the vessel, due to the viscosity.
In this case the higher temperature, during the process, is not collected by sensor 7 but sensor 3 with a maximum gradient between them of around 2°C. This is possible due to the flow created by the anchor different from the one in the water for the different characteristics of the fluids.

The heating system is stopped when the average temperature of the fluid is 64.5°C due to the large creation of the lather. The total time is 01:35:00 and at the end the temperature difference measured by sensor 3 and sensor 1 is around 2°C. In water case the total time for the same range is 01:25:00 with a homogeneous temperature of the fluid.

The cooling phase, with the same settings of the heating, is shown in figure 4.19.

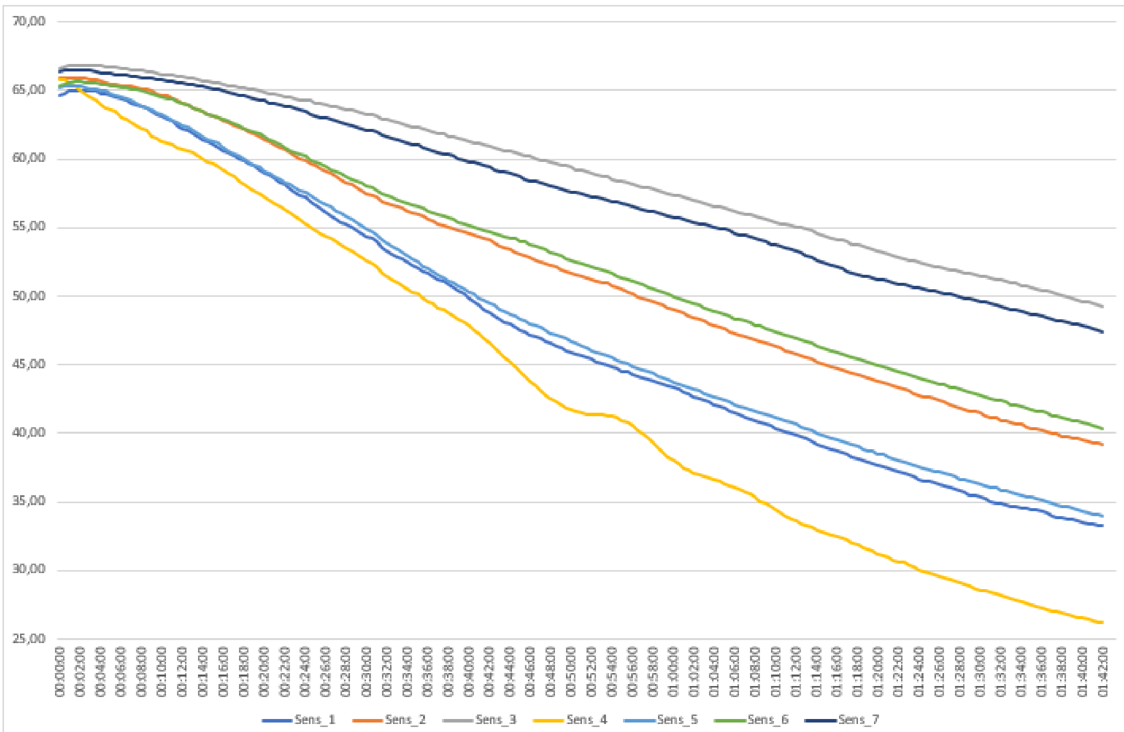

Figure 4.19. Cleanser with anchor speed of 2,5 rpm (cooling)

During the cooling phase, due to the half-pipe structure and the entry of cold water at the bottom, like in previously cases, the fluid on the bottom decreases its temperature faster than the top one. Differently from the water, a difference between the sensors at the same height is visible especially at the top. The sensors near the jacket have temperature lower than the sensor in the middle and the gradient between sensor 7 and 3 has a maximum equal to 2°C, while between sensor 6 and 2, the maximum is around 1.5°C.
Since the viscosity of the cleanser is higher than the one of the water, the temperature difference between the sensor 7 and 4 is higher with a maximum at around 38.5°C at the end, where the average temperature is 21.5°C. The higher viscosity leads to a different flow therefore a higher heat transfer coefficient.

The time from an average temperature of 66°C to 38.5°C is 01:42:00 while in the same range for water the time is 01:14:00 with a lower temperature gradient. Therefore, viscosity influences the heating or cooling time of the fluid in laminar flow and, as it increases the heat transfer coefficient, the temperature gradients inside the mixer increase.

4.5 Tests with emulsion

In this section, an emulsion product is produced and the data collected are shown. The temperature detected in the production cycle is shown in figure 4.20.

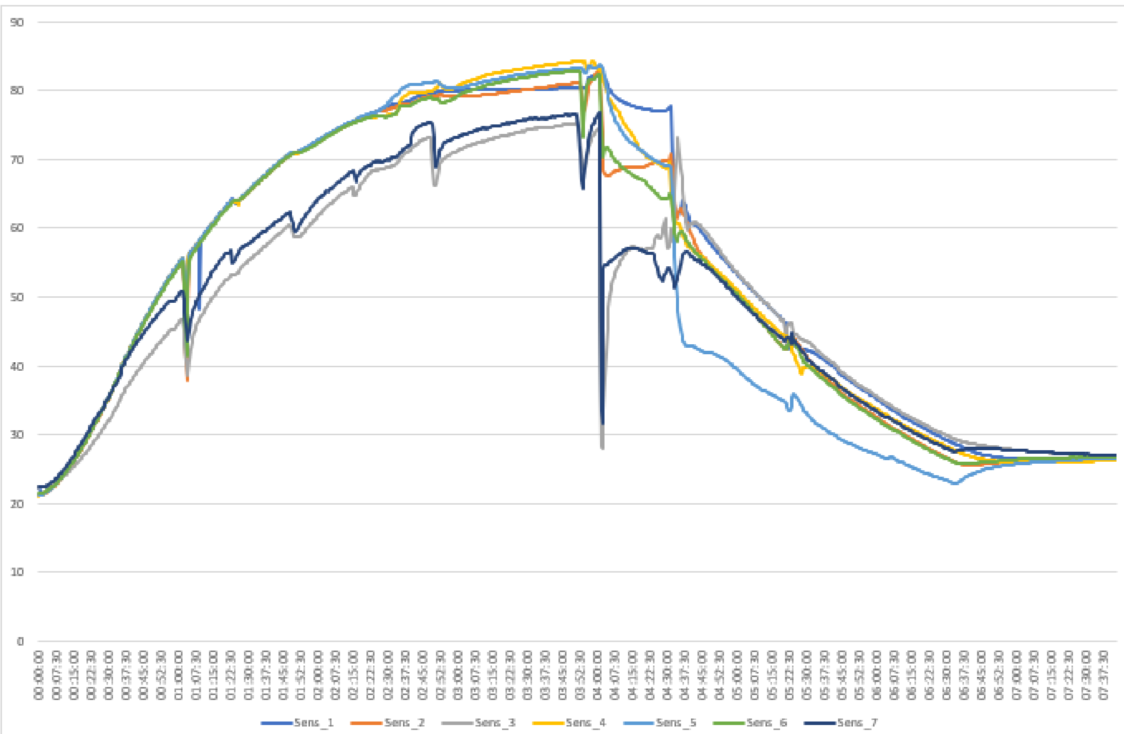

Figure 4.20. Emulsion production test

As a first step 48 kg of water is added with a material used for gelification and the heating system is set to 85°C. In this first part of production, sensor 3 and 7 are not immersed in fluid and the speed of the anchor is equal to 28 rpm while the speed of the propeller is around 500 rpm. At 01:04:00 4 kg of liquid raw material is added, leading to an immediately decrease of the temperatures of some sensors due to the lower temperature of the material added but after 1 minute, the material blends

and the temperature increases again. From this moment onwards, the propeller is stopped.
At 02:52:00, when the temperature of the whole fluid is included in 70°C and 80°C, the two powdered raw materials are added. The temperature decreasing steps of sensor 3 and 7 are caused by the opening of the glass window on the cover of the tank to observe the mixing process. The opening leads to a loss of hot air that is dispersed in the external environment. At 03:55:00 other raw materials are added, and the speed of the propeller is set to 300 rpm.
When the fluid in the mixer is homogeneous, the cooling phase starts, setting the temperature of the water, which flows in the half-pipe system, equal to 5°C. 19 Kg of water are added, causing a decrease in temperature of the top sensors because initially the water remains at the top of the fluid due to the different viscosity. Now, all sensors are immersed. Below 45°C, at 05:23:00 the last powdered raw material is added. The result is the product with viscosity equal to 4000 cps.

The collected data show the high temperature gradients in the mixing process. These differences in real industrial cases can lead to degradation or difficulty in a blending operation, especially for the big-sized industrial mixers.
As shown in the tests performed, the system is able to collect temperature data during a mixing process detecting the temperature gradients in a pilot mixer where the distances between the sensors are shorter than in the real industrial case. It is able to work in different conditions, indeed the temperature is collected indifferently in air or in viscosity fluid and the hardware can resist without any damages until 90°C. Moreover the system is able to work indifferently with any speed of the anchor and propeller. The accuracy and sampling frequency allow the usage of this system for fluid dynamics models to improve the process and the hardware involved.

Chapter 5

Conclusion

5.1 Future steps

The system tested in this work is only a prototype, indeed some modifications have to be applied with respect to all Good Manufacturing Procedure (GMP) requirements to obtain an industrial version of the system designed. The prototype can be used to collect data from a pilot mixer for the initial computational fluid dynamics simulations without modifications. In fact, before the realization of the industrial version, fluid dynamic simulations are performed to understand the benefits that the system may have in terms of costs, time and quality.
As far as implementation in industrial mixers is concerned, to have a limited number of cables, the sensors are soldered together like in figure 2.12. where only three cables are connected to the controller. The sensors' wires can be changed: it is possible to use smaller PTFE cables rather than the present ones. A possible choice of cables is the 28 AWG type, that means having a diameter of 0,321 mm. Another modification regarding the sensors is the metal cylinder which needs to be changed from stainless steel 316 to 316Lis in order to be chemically compatible with the all materials used. The metal cover can be changed or the sensor can be encapsulated in a 316L cover full of thermal conductors.

The box containing the Arduino board can be realised in PTFE with a 3D printer. The cover needs to respect all requirements about the cleaning and sanitation phases. The shape and dimensions will need to be in accordance to GMP requirements, and must also contain a battery pack with enough capacity to power the system for 2-3 years.
Battery life can be extended as it can be recharged with the use of vibrations, in fact some piezoelectric microstrip devices create a voltage difference due to the presence

of the vibrations. The piezoelectric transducers are more suitable as kinetic to electrical energy converters. In addition to the advantage of being smaller and lighter, the energy density produced is three times higher as compared to electrostatic and electromagnetic cases. A study on the vibration frequencies of the machine can be performed to discover better devices able to recharge the battery. These devices harvest a power around $100\mu W/cm^2$ and some of them are commercially available according to the resonant frequency.

In prototype, the user has access to the system through a computer connected to Arduino receiver board. This allows to display the results with the PLX DAQ tool, but this is not the only possible solution. Indeed other applications can be used or the board can be connected directly to a server using an Ethernet or a RS232 cable. To implement these protocols on the board, low cost shields can be integrated. This allows the system to control and save data on the company server tool. Another solution is that of implementing a wireless connection to the board like Bluetooth to connect the system to any mobile device or computer with the usage of cables.

5.2 Conclusion and results

The system respects design specifications, expect for the cleaning parameters, and the prototype realized works fine as expected. Its flexibility makes it a useful temperature measurement system for pilot mixers, where cleaning is operated manually. In future steps, the improvements to realize an industrial version are explained and can be easily implemented after a study on its shape and on the benefits that it could bring.
Nowadays, the system will be used to perform tests in the pilot mixers in order to collect data for fluid dynamic simulations. These data can help realize a more precise model of fluid and flow or to confirm the results reached by the simulation tool. Indeed as highlighted from the tests, the system works indifferently in air or viscosity fluid and it is able to detect the temperature gradients involved in the mixing process. The accuracy and the working sampling frequency allows its usage both as a possible control and as data collection for further studies.

The flexibility added to the low power consumption make this system a useful investigation instrument to understand the variation of temperature in the mixing process and it is a little step to the integration of sensors inside this kind of machine in order to collect data to control all parameters of the process and, thus, the quality of the product, but also to collect data to optimize the equipment or the process itself in terms of cost and time.

As far as the performed tests are concerned, they highlight a direct influence between the flow and the physical characteristics of the fluid and the temperature gradients during the heating and cooling phase, as expected. The flow is controlled with the speed of the motion system which includes the anchor and the propeller. In the water experiments, the minimum speed of anchor (2,5 rpm) is sufficient to reach a homogeneous temperature during the heating phase, while to obtain the same result in cooling phase, the speed of the anchor should be set to 15 rpm or equal to 2,5 rpm plus the propeller set to 320 rpm. Indeed the different systems to increase or decrease the temperature lead to different times and phenomena. The heating system made of a jacket full of thermal oil heated up with a resistance is better in order to obtain a homogeneous temperature during the phase, with the minimum consumption of energy for the motion systems, but it takes long time to reach the temperature setpoint. Instead the cooling system made of half-pipe structure works better as far as time is concerned but is worse in energy consumption.
The cooling system, which is the same as the one integrated in industrial mixers, highlights an issue connected to the temperature gradient in the mixer whenever the flow is not turbulent enough. Industrial mixers also use for the heating system the half-pipe structure and if the same results are reached in the heating phase, the problem of overheating, which leads to an increase of time and degradation of material, can happen without any control of the operator. In fact the only one sensor integrated in the mixers is not able to detect this phenomenon with a consequent loss in terms of costs for the company.

List of figures

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