Politechnika Wrocławska
Wydział Mechaniczny

Faculty of Mechanical Engineering

APPLICATION OF DESIGN OF EXPERIMENT TOTAL QUALITY MANAGEMENT TOOLS IN COMPOSITE MANUFACTURING SYSTEM - CASE STUDY - RESIN CASTING PROCESS OPTIMIZATION

FINAL DEGREE PROJECT

Author: Adrian Ferrer Lafarga

Supervisor:
Prof. Grzegorz Lesiuk

Supervisor:
Pascual Guillamon, Manuel

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ABSTRACT

Design of experiments is a statistic tool that is used when you are interested in the effect that one or more factors cause in the results of experiments. This method allows to make less experiments that are supposed to do but obtaining the same results.

In this project, this tool would be used for making an optimization on the resin casting process, analysing some parameters. Therefore, the goal of this project is to achieve the best combination of parameters on the resin casting process of composite materials.

The actual project is realized by the mechanical engineering student Adrian Ferrer Lafarga, as final degree project after approving the courses that the degree is composed.

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CHAPTER I

1. Introduction to Design of Experiments - Taguchi method

Design of experiments is a statistic tool that is used when you are interested in the effect that one or more factors cause in the results of experiments. This statistic tool allows the user to save a high percentage of experiments that should be done in order to optimize the parameters of an experiment.

This method was developed by Ronald Fisher, that apply his statistic knowledge to agriculture on the XX century. As this method is suitable for many types of experiments, it can be applied to many fields such as industry and medicine but also in other fields that has no relationship with science as marketing.

There are many types of design of experiments but in this project the method that we are going to apply is *Taguchi method. Taguchi method* refers to the statistical method developed at the beginnings of 80s that, Gen'ichi Taguchi, a Japanese engineer developed for quality control in industry. The philosophy of this engineer was about all surrounding the quality of the product. For him, quality was defined as the lose that a product makes in the society, as lower lose, better quality.

The utility of this method is that is apply to experiments that have a great number of factors with different levels, that supposed a big number of experiments to be conducted. Applying this method simplifies a lot the obtention of results as compared to normal obtention. In this project, we have 3 factors with 3 levels each one. The combination of this factors gives us to conduct 27 experiments but, applying *Taguchi method,* we obtain that, on a combination of 9 experiments, we can determine how affect each factor to our results, determining then which is the best combination of the levels of each factor.

For determining which experiments would be conducted and which not Taguchi method applies an orthogonal matrix, which lets us obtain the combination of factors and their levels for obtaining the experiments that have to be done in order to get the results.

In order to understanding how *Taguchi method* works I will give an example. This example would be the recipe of a cookie. We have three factors, the percentage of milk the cookie has, the time that it is baked and the temperature for baking it. We want to make as fast as possible but remaining to be with a good taste. It is stablished three levels of each factor[1], the percentage of milk can be 0%, 30% or 50%, the temperature of the oven 80°C, 87.5°C or 95°C and the cooking time time 1, 2 or 3 hours.

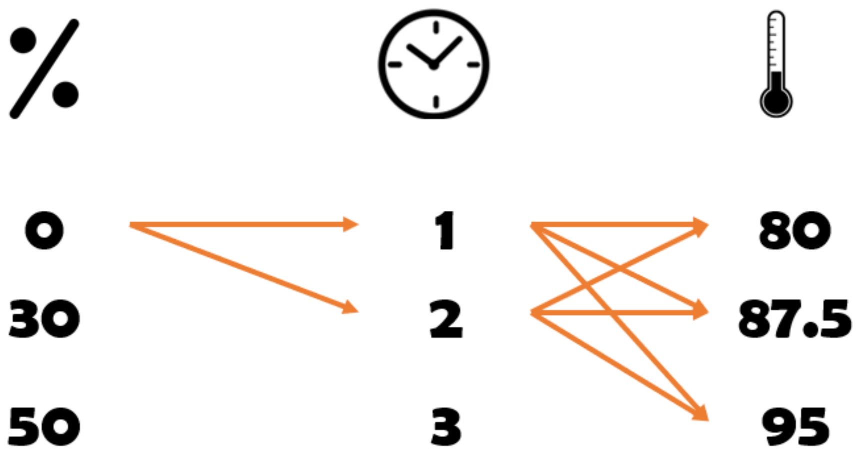

Figure 1 - Taguchi method example

So, we have 3 factors with three levels, for making all the types of cookies that can be done we have to bake 27 different cookies. But what happens if we apply *Taguchi method*? We would only have to make 9 different types of cookies, obtaining the best combinations looking as how affect each factor to our result and choosing the one that have the best relationship with both results that we expect, the time and the taste.

2. Design of Experiment - Resing Moulding

The method that is explained before is going to be applied to epoxy resin. This resin is used to make the fiberglass. Fiberglass is a composite material made by glass filaments of a small diameter that are joined by a resin. These filaments of glass have a good behaviour against tensile stress and withstand a great deformation, although are not good at compression. The resin has a good behaviour at compression, what results in a material with good mechanical properties at a low weight and a low price.

The resin is going to be used for this purpose is epoxy resin, the most resistant adhesive used nowadays. Specifically, is the *Biresin CR141*, a resin that is prepared with the mixture of three different components, A that is the resin[3], B that is the hardener[3], and C that is the accelerator[4]. The proportion of the components the manufacturer give us is not going to be changed, we would use the one that is specified in the product data sheet. This proportion is 100 grams of A, 90 grams of B and 2 grams of C.

Figure 4 - Component A

Figure 3 - Component B

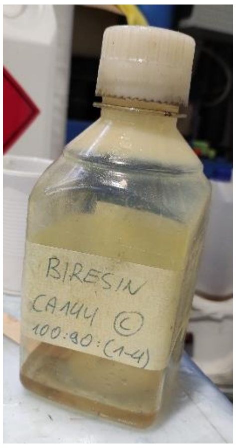

Figure 2 - Component C

The components A and B of the *Biresin* are contained in metallic barrels as the most part of the mixture is made by these components. The component C is given in a small quantity.

The fiberglass is going to be used for the reinforcement of concrete, for this reason is important that the resin be as non-flammable as possible as it is used in buildings. For this purpose, we are going to add flame retardant to the mixture. The problem of the flame retardant is that affects to the other mechanical properties of the resin so we would have to find the best proportion to do not affect too much to the stress that resin supports but at the same time to do not burn too easily.

In addition, we would try to make the process the fastest as possible and with a lower temperature to save energy. Attending to this, the parameters in the process that we are going to change are three, so we would have three factors in our *Taguchi* matrix. These parameters are the following ones:

- **Temperature**: The resin, according to the manufacturer, must be cured 3 hours at 80 degrees, 3 hours at 120 degrees and 3 hours at 140 degrees. 139 degrees is the glass transition temperature of this resin, which is the temperature when a polymer goes between the rigid and a flexible state. For this, in the experiment we are going to use three combinations of temperature based in the manufacturer sequence of temperatures. One combination would be 7.5 degrees more in each state, one the manufacturing temperature and the other 15 degrees more, so we would see if go upper this temperature *tg*(glass transition temperature) get us a better results or makes faster our production of the resin. Considering this, the levels would be:
    - 80°C, 120°C, 140°C
    - 87.5°C, 127.5°C, 147.5°C
    - 95°C, 135°C, 155°C

- **Time**: This process is very time consuming so we would try to reduce it. Instead of having 3 hours for each phase we would try with one and two hours less, so we would have 3+3+3, 2+2+2 and 1+1+1, so we would see how affects to reduce this time on the mechanical properties. The less time we consume more optimized would be our process. The levels would be:
    - 1 hour, 1 hour, 1 hour
    - 2 hours, 2 hours, 2 hours
    - 3 hours, 3 hours, 3 hours

- **Flame retardant**: To the mixture of resin we would add flame retardant which is a substance that prevents or inhibits the outbreak of fire. We would use the following proportions: 0%, 30% and 50%. This proportions are above the resin we have, not the whole mixture, for example, if we have 50%, for 100 gr of A, B and C mixture we would add 50 grams of flame retardant. The goal is to have the greatest proportion of flame retardant without affecting the maximum stress that supports the resin. The levels are the following:
    - 0% of flame retardant
    - 30% of flame retardant
    - 50% of flame retardant

Calculation of factors

After defining the factors and levels we have 9 factors and 9 levels, which results in 27 combinations and therefore 27 experiments. As it would be time consuming and very costly it is applied the *Taguchi* matrix, which results that with 9 experiments we could guess the best combination of the factors to our purpose.

The *Taguchi* matrix can be calculated with statistics professional programs as the high used *Minitab*[5]. Introducing the data that we decided to analyse print us the combinations that we must use and, when having the results, show us how each parameter affects the results.

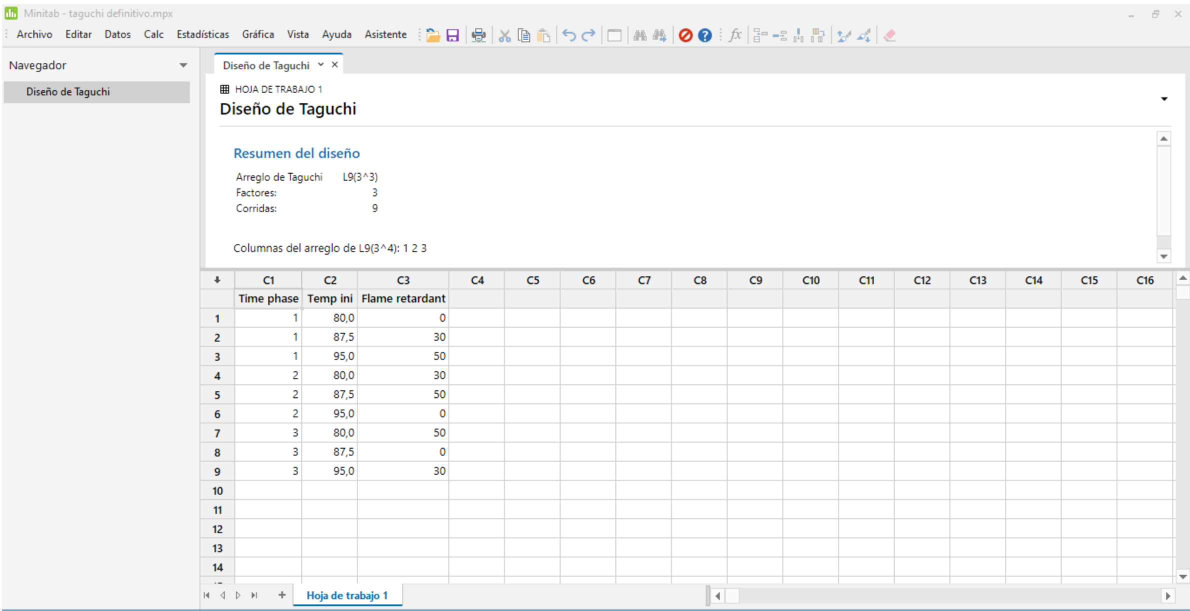

Figure 6 - Minitab interface 1

For using a Taguchi method, we have to select in the top tool bar Statistics→DOE→Taguchi→Create Taguchi design

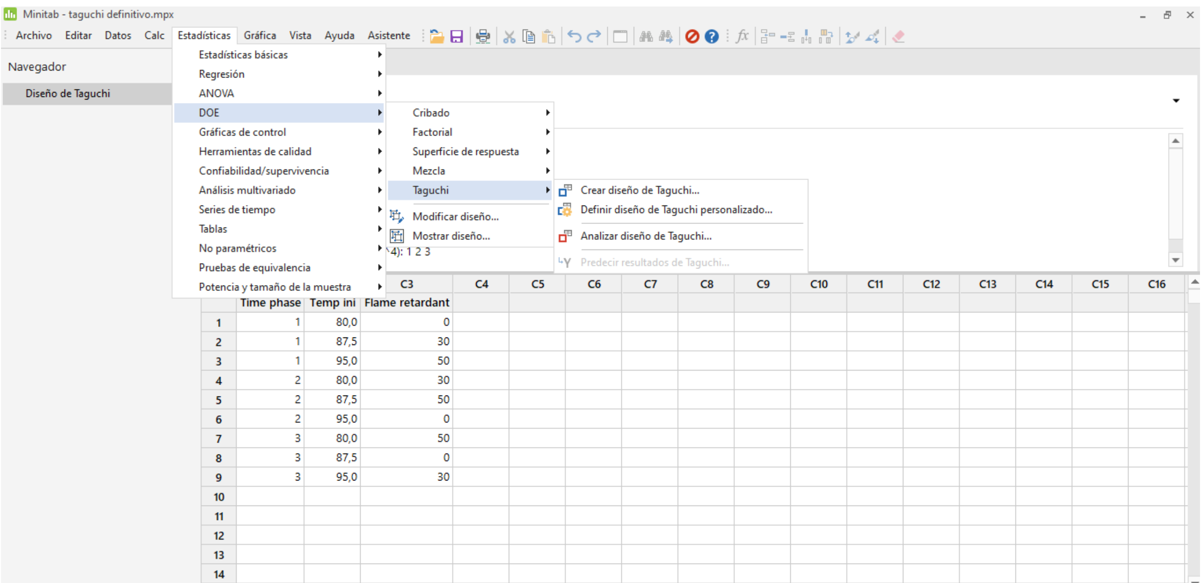

Figure 5 - Minitab interface 2

Then we must select the number of levels and factors our matrix would have.

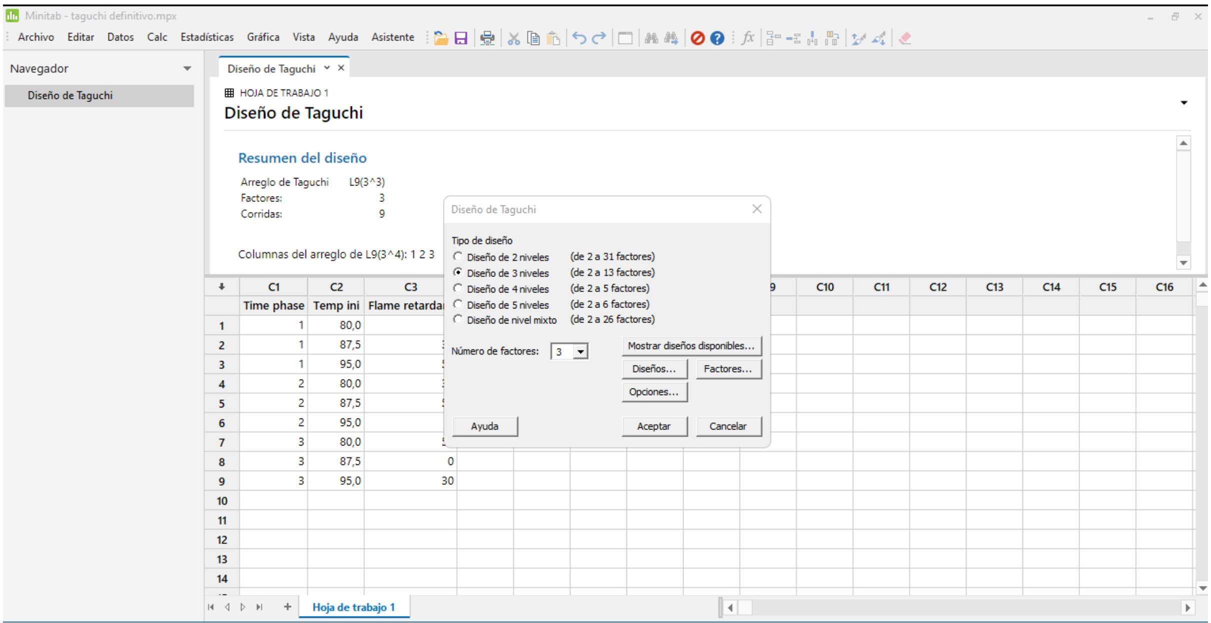

Figure 8 - Minitab interface 3

And then click in Designs and select in this case L9.

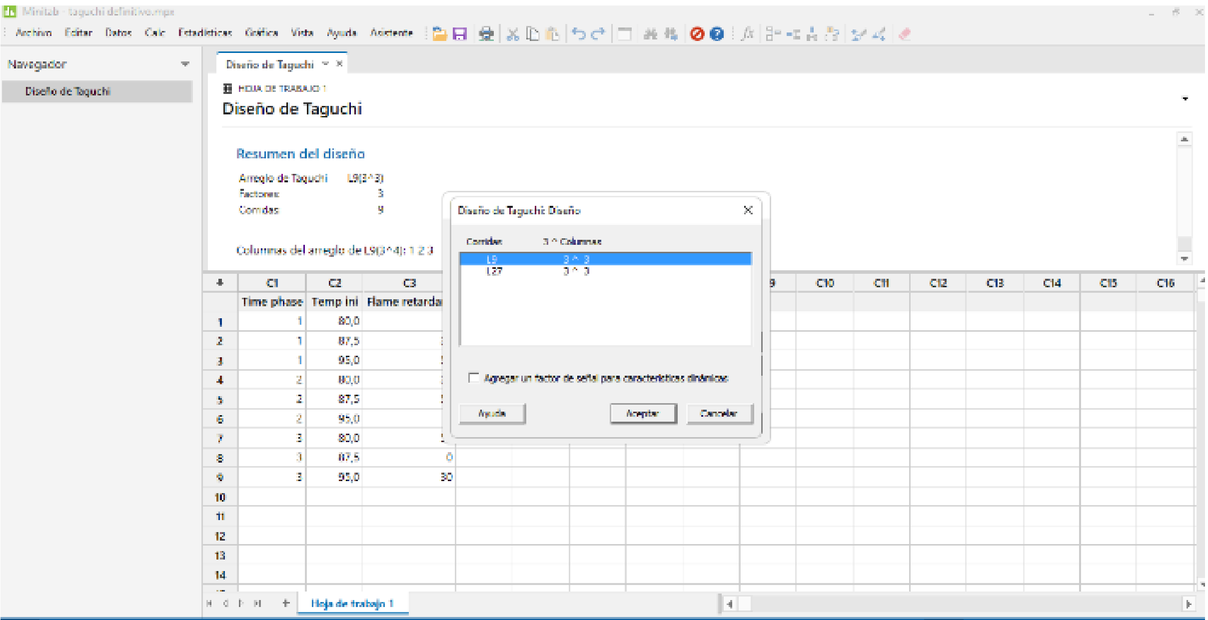

Figure 7 - Minitab interface 4

Finally introduce the parameters that we are going to look for in the experiment:

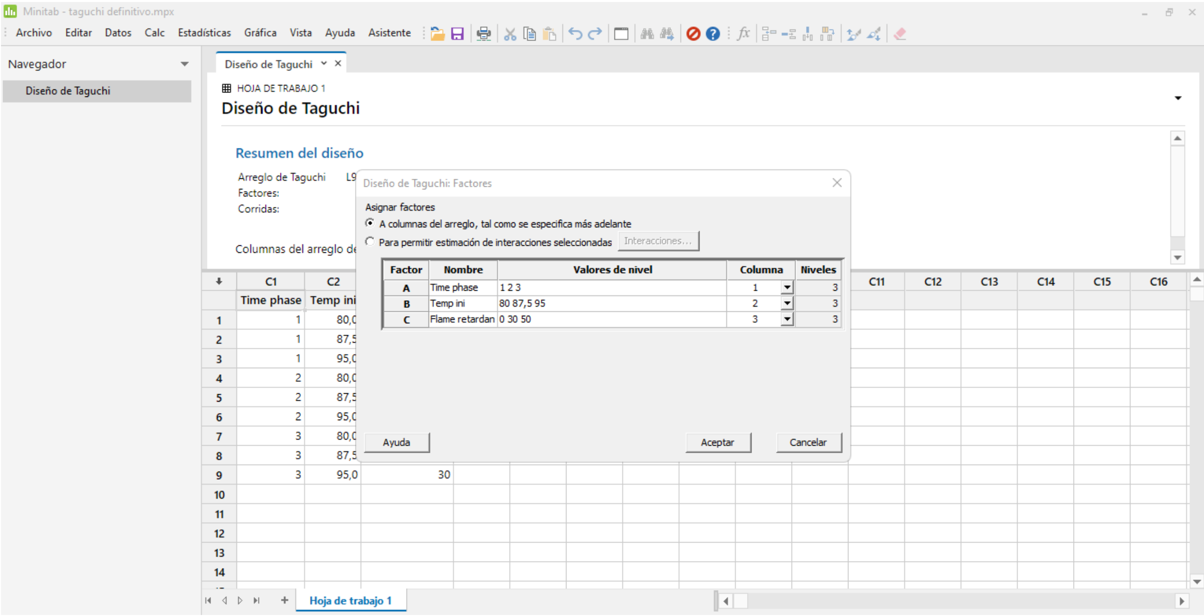

Figure 9 - Minitab interface 5

And we obtain the combinations that we have to conduct in order to get the results:

	Time phase	Temp ini	Flame retardant
1	1	80,0	0
2	1	87,5	30
3	1	95,0	50
4	2	80,0	30
5	2	87,5	50
6	2	95,0	0
7	3	80,0	50
8	3	87,5	0
9	3	95,0	30

Figure 10 - Experiments to be conducted

3. Manufacturing

For the purpose of testing the mechanical properties of the resin is going to be moulded three

Figure 11 - Samples mould

pieces of each mixture in order to do the experiments. These pieces have the following size: 90*20*10 mm. The mould[11] used is made by rubber.

For producing the resin we start with a plastic glass[12] to make the mixture. This plastic glass should be flat, without setoffs in the walls. We use a scale to measure the components. First of all we add A component, that has a high viscosity, therefore is hard to handle.

Figure 12 - Glass with resin in the scale

Then B component, which is more liquid and easier to add. We mixture both with a wooden stick with rounded tip, softly and without hitting the walls for do not make many air bubbles. Finally, we add C and mix too. In the case of the C component, we have to add very few, and it has to be done precisely, because we can alter a lot the mixture only adding one gram overly, so we use a syringe to add this component. C component has a similar behaviour as water. In case of preparing with flame retardant we add it after mixing C. This flame retardant is presented powdered, it is easy to add.

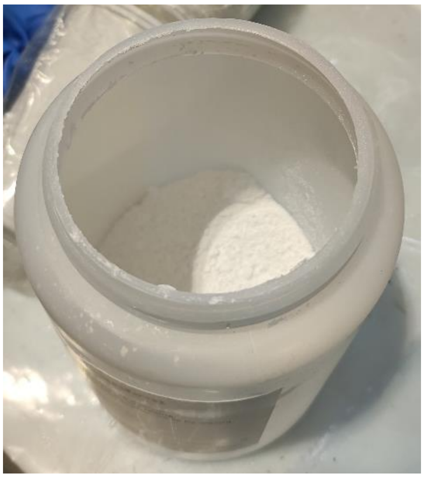

Figure 13 - Flame retardant

When the resin is mixed with the flame retardant, it turns white instead of being a translucent yellow[14].

Figure 14 - Resin with flame retardant

Before cured it in the oven we must eliminate the air bubbles of the mixture for do not affect the mechanical properties of the piece. For this we use a vacuum chamber[15/16]. We must take into account that when the mixture has flame retardant it is more costly to eliminate the bubbles and if we spend too much time it can be evaporated some components that could weaken the component. We should vacuum until the foam stops to go up.

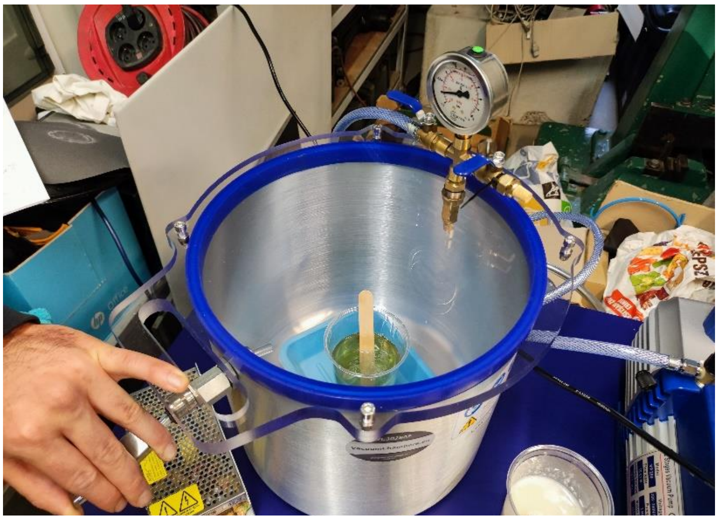

Figure 16 - Vacuum chamber 1

Figure 15 - Vacuum chamber 2

In the process of vacuum the air of the resin two different models of vacuum chamber[15/16] has been used. After vacuum we put the mixture in the mould, introduce into the oven[17/18] and let it cure.

The ovens can be programmed to follow the sequence of temperatures and time we need to cure the resin, therefore is only necessary to pick the samples after the process is completed. In the process two types of ovens has been used[17/18].

After the samples are cured, we must prepare it. We have to sand the surfaces of the samples until are flat. Specially the ones that have the most part of air bubbles, because is part of the material that do not have the same mechanical characteristics.

Figure 17 - Oven 1

Figure 18 - Oven 2

The difference between both parts can be appreciated as can be seen in the following picture:

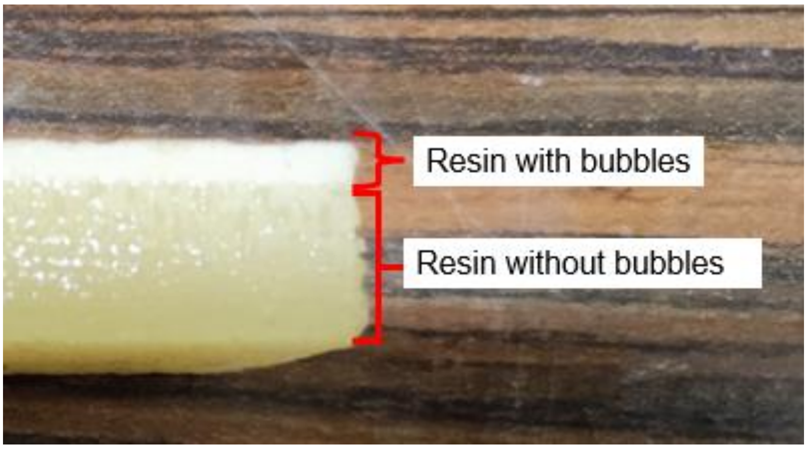

Figure 19 - Sample of resin with flame retardant

Then we make a notch[20] for getting sure the crack in the test would happen in the middle of the piece.

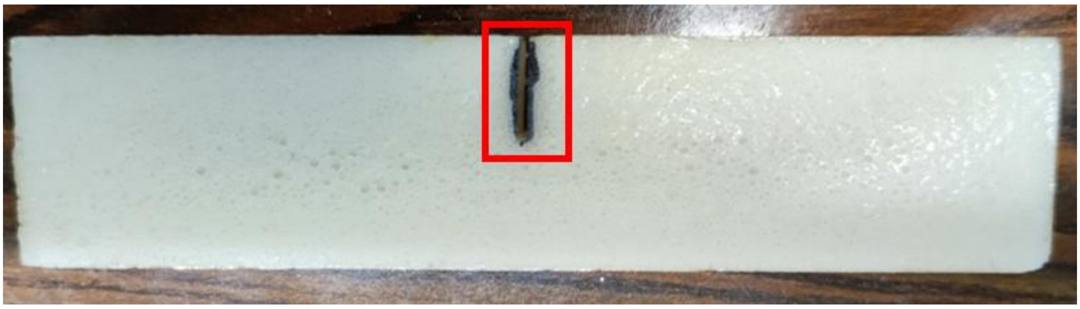

Figure 20 - Sample with notch

For sand the surfaces we use the following sanding-polishing machine[22], cooled by water[21]

Figure 22 - Sanding-polishing machine

Figure 21 - Sand process

to not increase too much the temperature in the process.

Problems

After making the samples it has been detected some situations that could affect the results.

One problem is that has been used different ovens on the process, as well as different vacuum chambers, what could make different quality of the resin in each sample. Also, for the resin of some samples a remover has been used to remove the bubbles in the vacuum process while in others not.

The resin has been prepared in similar weather conditions so we should not have problems with that, although have been prepared a little bit spaced on time considering the curing of the first and the last sample.

Another issue happens when the mixture has flame retardant as is very costly to eliminate the air using the actual vacuum chambers. This bubbles that are remaining in the piece could affect the results, showing that the mixture with flame retardant have worse results as it would have without the air but, as far as we cannot do anything more, we have to take into account that using flame retardant will produce more air in the final product and therefore a worse mechanical properties.

4. Testing

For the testing of the resin, we use three samples of each mixture. First, we measure the size of the samples, width and thickness. We use a fracture test, with two points of support and one point in the middle applying the pressure[23].

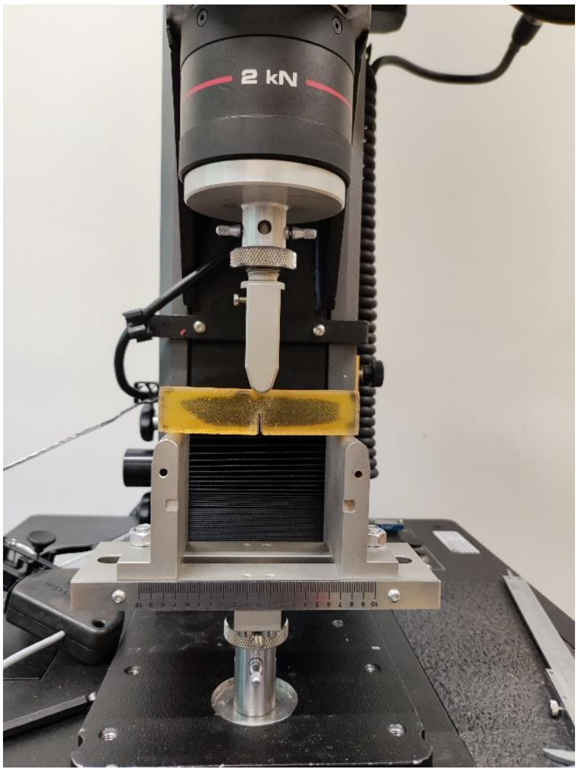

Figure 23 - Test machine

With the fracture test we obtain the maximum force applied to the sample. Then we measure the crack length[24] for obtaining the stress that our sample support.

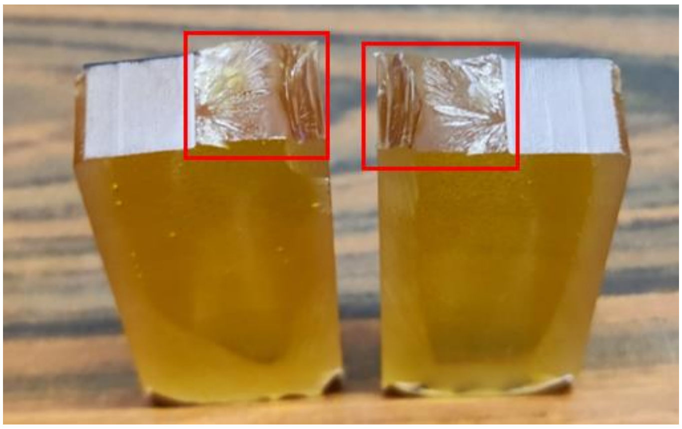

Figure 24 - Crack lenght(sample without flame retardant)

Figure 25 - Machine control

For preparing the test first we turn on the computer program[26] that controls the machine. Then we put the sample in the two point support, with the notch face down, as it is shown on the picture[23]. With the machine control[25] we adjust the high of the pressure point. We must put as closer of the sample as possible. For this we press the button that is highlighted in the picture[25] for getting sure the machine is not going to overpass 5 kN. Then we use the wheel for get down the pressure point, until the display shows us that is making some pressure in the sample when we scroll down. Finally we start the test from the computer.

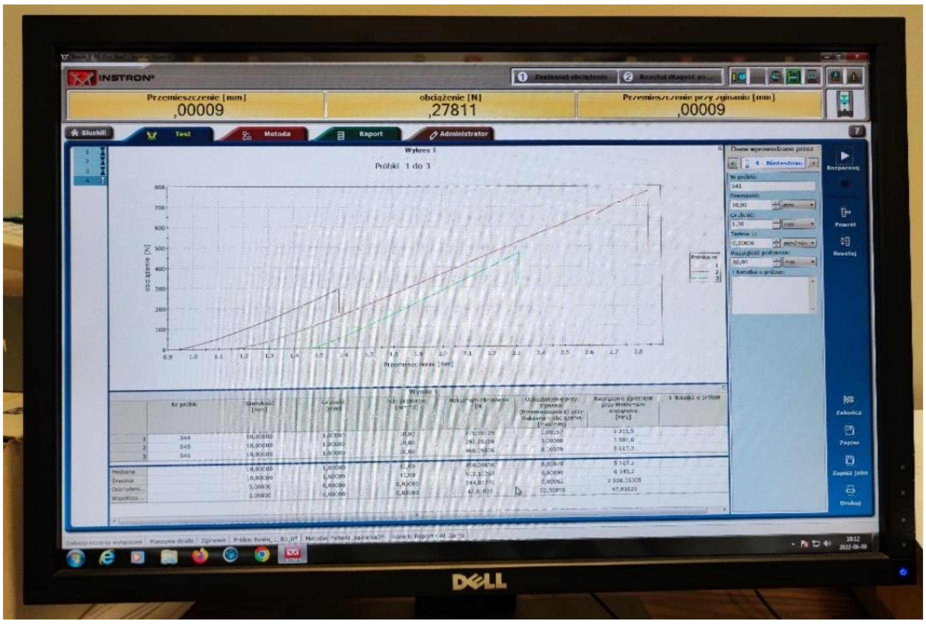

Figure 26 - Test computer program

The computer program[26] shows us the force applied and a graphic with the force and deformation of the sample, compared with the previous test that are done.

5. Data analysis

For the data analysis we use as input the maximum force of the test and the width, thickness and crack length of the sample for obtaining the fracture toughness, which is the value that we are going to compare between the different processes of resin making, for knowing which is the most suitable. The results obtained are the following ones:

Chart 1 – Experimental results

Material	Thickness B	Width W	Crack length a	Force [N]	KI
1,80,0 S45	10,1	20	11,3	292	3,123
1,80,0 S41	10,2	20	10,8	468	4,515
1,80,0 S42	10	20	11	358	3,654
2,80,30 S31	10	20	10,5	212	1,978
2,80,30 S32	10	20	12,5	218	3,012
2,80,30 S33	9	20	10,75	264	2,861
3,95,30 S22	8,5	20	11,2	210	2,618
3,95,30 S21	9,4	20	11,6	415	5,055
3,95,30 S23	9,6	20	11,1	385	4,171
1,88,30 S12	10,8	20	11,4	301	3,069
1,88,30 S15	9,9	20	13	300	4,692
1,88,30 S13	10,7	20	10,6	278	2,467
2,95,0 61	9,3	20	8,6	357	2,640
2,95,0 62	11	19,7	7,7	262	1,498
2,95,0 63	10,6	20	8,4	345	2,174
2,88,50 71	9,5	20	7,8	242	1,563
2,88,50 72	9,4	20,4	8,5	188	1,284
2,88,50 73	9,5	20	8,7	201	1,477
1,95,50 81	9,5	20,3	8,4	195	1,317
1,95,50 82	9,5	20,2	9,5	195	1,572
1,95,50 83	9,5	20	7,5	199	1,233
3,88,0 91	10	20	8,6	284	1,953
3,88,0 93	8,8	19,8	8,1	495	3,697
3,88,0 94	10	20	8,6	288	1,981
3,80,50 101	8,6	20	11,1	213	2,576
3,80,50 103	9,5	20	11,6	411	4,954
3,80,50 105	9,4	20	11,2	399	4,498

The name code is the following one:

1_80_50

TIME TEMP %

In this case it would be 1-hour phases, 80 degrees of starting temperature and 50 as flame retardant percentage. Then we have the code of the sample that is analysed. Instead of 87,5 degrees we have 88 because the oven can not use decimals on the temperature, so the number was rounded.

We expect the maximum KI with the lower cost of production, and the highest quantity of flame retardant. The KI parameter is calculated following this equation:

$$K_{I} = \sigma_0 F_{I} \sqrt{\pi a}, \; K_{II} = \sigma_0 F_{II} \sqrt{\pi a}; \; \sigma_0 = \frac{3}{2} \frac{PL}{W^2 B} \quad (4)$$

Equation 1 - KI equation

The KI parameter, stress intensity factor mode I, is one of the most important parameters of materials under stresses. This is because this parameter is able to describe all the stress field in the crack area. For this reason, we would use it to compare the different samples. As we made three test of each mixture, we would use the arithmetic average to obtain only one value to apply *Taguchi*. Applying the average we obtain the following results:

Material	KI
1,80,0	3,764
1,88,30	3,409
1,95,50	1,374
2,80,30	2,617
2,88,50	1,441
2,95,0	2,104
3,80,50	4,009
3,88,0	2,544
3,95,30	3,948

Chart 2 - Average experimental results

After analysing the results, we obtain the following graphics:

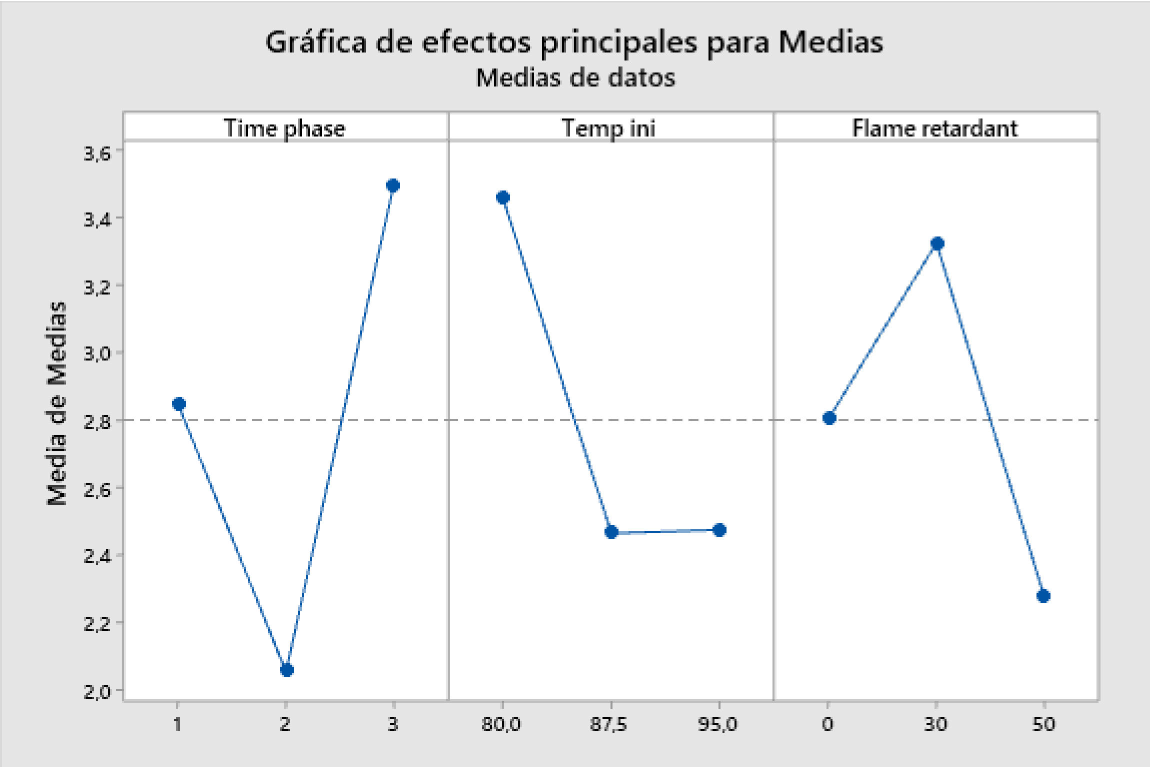

Figure 27 - Effects of the parameters on KI

The graphics[27] shows us how each individual parameter affects our KI. We are going to analyse each parameter.

Time

On this parameter we look for the less time possible wasted in the process but without affecting too much the resistance that our resin has. In this graphic should show a line increasing or decreasing instead of a line going down and then up but, as it is shown is better to cure the resin only one hour or three depending on the requirements, we have but never two. This may occur by the problems explained before in the process of curing the resin. Although this, the graphics show that we can have the **77,78% of the stress that our resin supports by only using the third of time curing**, 3 hours instead of 9 hours the producer tell us.

Temperature

On this parameter it is not beneficial to increase the temperature the producer tell us, as the graphics show. For this, **we should not elevate the temperature**, as we would wasting energy and making a worse product.

Flame retardant

Flame retardant should make worse the properties of the resin, but our graphics show that is better to use a thirty percent of flame retardant instead of zero. Probably these results are because the problems of the process of making the resin, although this we can say that we **can use till 30% of flame retardant without making worse the properties of our resin**.

Results

Attending to what it is said, we should use the sequences of temperature that the maker tells us, 80°C, 120°C, 140°C, use 1 hour per phase or 3 hours if we require the 22% of extra resistance and flame retardant till 30% in the process of resin curing for obtaining the best results.

6. Conclusions

Taguchi method and other Design of Experiment methods are very useful for saving resources when we must conduct a large number of experiments in order to optimize the parameters of a product. Although this we must take into account that these experiments have to be recreated very precisely and with the same conditions because if we conduct all the experiments one error could not be very important but, if we use DOE for making less experiments an error has a very important impact on the results.

Attending to the results obtained we can deduce that may be in an experiment mistakes can be made, although this we could optimize the process, obtaining that with less time curing the resin it was not too much affected and that we could add a high quantity of flame retardant without affecting the properties of the resin. In addition, I have to say that the values of the maker give us where optimized correctly attending to the results obtained.

Finally say that I am thankful for carry on this project because I find it very interesting and useful for carry out tasks as engineer in the future.

CHAPTER II

1. Budget of the project

In this point it would be analysed the costs of developing the actual project. Some prices are an estimation, therefore the values that are displayed can be variable. Despite of this, this budget can give an approximate idea of the real cost of the project.

1.1. Material costs

Here we consider the price of the epoxy resin used. We made 9 experiments with 3 samples each experiment. For each experiment, considering the waste and the samples that were discarded, we use approximately 150 grams of resin and 35,5 grams de flame retardant per experiment. Therefore, we used 1350 grams of resin and 320 grams of flame retardant.

	Quantity	**Cost/Unit**	**Total cost**
Biresin CR141	1,35	80 zl	108 zl
Flame Retardant	0,32	264 zl	84,48 zl
			192,48 zl

1.2. Machinery costs

In this point, the machinery cost that is used producing the resin will be analysed. In the process is needed a precision scale for the resin components mixing, an oven for the cure and a vacuum chamber for eliminate the air from the mixture.

	Quantity	**Cost/Unit**	**Total cost**
Material	1	110 zl	110 zl

Oven	1	264 zl	4400 zl
Vacuum chamber	1	600 zl	600 zl
			5110 zl

1.3. Personal setup costs

Finally, it is considered the own setup, including the technological software and hardware used. The licenses used are student licenses, but the prices displayed are the full cost prices. In the process is used a laptop, the statistical software and the office automation software.

	Quantity	**Cost/Unit**	**Total cost**
Laptop	1	1000 zl	1000 zl
Minitab Software	1 (year)	4758 zl	4758 zl
Microsoft Office Software	1 (year)	1593 zl	1539 zl
			7297 zl

1.4. Total costs

As summary of costs we have that the global budget quantity is the following:

	Total cost
Laptop	192,48 zl
Machinery	5110 zl
Personal setup	7297 zl
	12599,48 zl

CHAPTER III

1. References

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2. Additional documents

Product Data Sheet
Version 04 / 2021

Biresin® CR141
Composite resin system for heat curing

Product Description

Biresin® CR141 is a three component, anhydride cured, low viscosity epoxy resin system suitable for the production of high performance fibre reinforced composites.

Application Areas

Biresin® CR141 system is particularly suited to the filament winding and pultrusion processes due to its low viscosity, good fibre wetting capabilities and very long potlife.

Features / Advantages

- The reactivity of the system can be adjusted by modifying the level of the accelerator (C) CA141
- Fast infiltration of dry fibres due to good wetting characteristics, low mixed viscosity and an elevated processing temperature
- Good wet-out of fibres, fabrics and non-wovens due to low viscosity and good wetting characteristics
- Approved by DNV GL - Certificate No. TAK00001AB

Physical Data		Resin (A)	Hardener (B)	Accelerator (C)
Individual Components		**Biresin® CR141**	**Biresin® CH141**	**Biresin® CA141**
Mixing Ratio, parts by	**Weight**	100	90	2
Mixing Ratio, parts by	**Volume**	100	87	2.4
Colour		translucent	transparent	amber
Viscosity, 25°C	mPa.s	~8,250	~40	~200
Density, 25°C	g/ml	1.16	1.20	0.98
			Mixture	
Potlife, 100 g / RT, approx. values		h	> 24	
Mixed viscosity, 25°C, approx. values		mPa.s	600	

Processing

- The material and processing temperatures should be in the range 18 - 35°C.
- The mixing ratio must be followed accurately to obtain best results. Deviating from the correct mix ratio will lead to lower performance.
- Before demoulding precuring of at least 2 h at 90°C is recommended.
- The final mechanical and thermal values are dependent on the applied postcuring cycles.
- It is recommended to clean brushes or tools immediately after use with Sika Reinigungsmittel 5.
- Additional information is available in "Processing Instructions for Composite Resins".

Mechanical Data, neat resin specimen approx. values after 3 h / 80°C + 3 h / 120°C + 3 h / 140°C			
Biresin® CR141 resin (A)	with **Biresin® CH141** hardener (B) and **Biresin® CA141** accelerator (C)		
Tensile strength	ISO 527	MPa	78
Tensile E-Modulus	ISO 527	MPa	3,200
Elongation at break	ISO 527	%	3.3
Flexural strength	ISO 178	MPa	145
Flexural E-Modulus	ISO 178	MPa	3,100
Compressive strength	ISO 604	MPa	122
Density	ISO 1183	g/cm³	1.20
Shore hardness	ISO 868	-	D 87
Impact resistance	ISO 179	kJ/m²	18

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Postcuring

The suitable cure cycle and the attainable mechanical and thermal values depend on various factors, such as laminate thickness, fibre volume, reactivity of the resin system etc.
An appropriate cure cycle could look as follows:

- Heat-up rate of ca. 0.2°C/Minute until approx. 10°C below the required glass transition temperature (Tg)
- Followed by a dwell at that temperature of between 2 and 12 hours.
- Part(s) should then be cooled at ~0.5°C per minute

The specific postcure should be adapted to the required technical and economic requirements.
To measure the mechanical performance of the resin system a Sika Advanced Resins standard cycle is used to ensure that the full Tg potential of the system in question is reached.

Typical Thermal Properties of Cured Neat Resin, after 3 h / 80°C + 3 h / 120°C + 3 h / 140°C

Biresin® CR141 resin (A)	with **Biresin® CH141** hardener (B) and **Biresin® CA141** accelerator (C)		
Heat distortion temperature	ISO 75B	°C	137
Glass transition temperature	ISO 11357	°C	139

Packaging (net weight, kg)

Biresin® CR141 resin (A)	1,000	220		10
Biresin® CH141 hardener (B)	1,100	220		9
Biresin® CA141 accelerator (C)			10	0.2

Storage

- Minimum shelf life of Biresin® CR141 resin (A) is 24 month and of Biresin® CH141 hardener (B) and Biresin® CA141 accelerator (C) is 12 month under room conditions (18 - 25°C), when stored in original unopened containers.
- After prolonged storage at low temperature, crystallisation of resin (A) may occur. This is easily removed by warming up for a sufficient time at a minimum of 60°C.
- Containers must be closed tightly immediately after use. The residual material needs to be used up as soon as possible.

Health and Safety Information

For information and advice on the safe handling, storage and disposal of chemical products, users shall refer to the most recent Safety Data Sheet (SDS) containing physical, ecological, toxicological and other safety related data.

Disposal considerations

Product Recommendations: Must be disposed of in a special waste disposal unit in accordance with the corresponding regulations.
Packaging Recommendations: Completely emptied packagings can be given for recycling. Packaging that cannot be cleaned should be disposed of as product waste.

Value Bases

All technical data stated in this Product Data Sheet are based on laboratory tests. Actual measured data may vary due to circumstances beyond our control.

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Legal Notice

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Further information available at:

Sika Deutschland GmbH
Subsidiary Bad Urach
Stuttgarter Str. 139
D - 72574 Bad Urach
Germany

Tel: +49 (0) 7125 940 492
Fax: +49 (0) 7125 940 401
Email: tooling@de.sika.com
Internet: www.sika.com

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