JOURNAL OF TRANSLOGISTICS

DOI: 10.37190/JoT2023_05

Grzegorz LESIUK*, Paweł ZIELONKA, Kayode OLALEYE,
Japhet SOOGABBE, Adrian Ferrer LAFARGA,

APPLICATION OF TAGUCHI METHODS (DOE) IN COMPOSITE ENGINEERING

Keywords: *composite, Taguchi method, flame retardant, epoxy resin*

In the paper, a design of experiment (DOE) in terms of the Taguchi method was used for the optimization of the manufacturing process. This approach makes it possible to do fewer experiments while still getting the desired outcomes. This research programme will use this tool to optimize the resin casting process by monitoring manufacturing parameters like temperature, curing time, and flame-retardant concentration. Thus, this study aims to determine the ideal set of parameters for the resin casting of composite materials. Based on experimental results, it was possible to find crucial factors and their influence on the fracture strength of resin for composite manufacturing systems.

1. INTRODUCTION TO DESIGN OF EXPERIMENTS – TAGUCHI METHOD

Thermosetting polymers cured with epoxy resins offer a wide range of uses due to their mechanical and physical characteristics; they are mostly used as matrix in advanced composites for automobile and aeronautical applications[1–4]. The following are some of these materials' primary characteristics: Outstanding adhesion to a wide range of substrates; (ii) excellent resistance to chemicals, especially in alkaline environments; (iv) minimal shrinkage; (v) beneficial electrical insulation properties; (vi) excellent corrosion resistance; and (vii) ability to cure over a wide temperature range are just a few of the material's many outstanding qualities [2–5]. Understanding the kinetics of epoxy resin curing can help you optimize composites' properties, reducing processing times, energy usage, expenses, and product quality [4–7]. Because it affects the final qualities of fabricated parts, the epoxy resin curing process is therefore the most important step in the manufacturing process. Epoxy resins undergo cross-linking or curing processes that create a three-dimensional infusible network by reacting hydroxyl groups or oxirane functional monomers. Both direct coupling of the resin molecules by a catalytic homopolymerization and

* Faculty of Mechanical Engineering, Wrocław University of Science and Technology.

coupling through an intermediate reactive known as a curing agent—compounds containing active hydrogen, such as polyamines, polyacids, polymercaptans, and polyphenols, to name a few—can cause the cross-linking process [3,4,8,9]. The reaction is extremely exothermic due to the high degree of reactivity of chemical species involved in the curing process; hence, the polymerization of epoxy resins must be carefully regulated to avoid the reaction heat being released from the process accelerating itself [2–4]. It has been shown that a number of analytical methods can be used to track the reaction's intensity and ascertain the epoxy resins' degree of curing [1–4,10–12]. Fourier-transform infrared spectroscopy (FTIR) is one of the direct methods that may be used to assess the concentration of one or more reactive groups over time (during the curing process). The main reasons FTIR is employed are its nondestructive approach, practicality, and adaptability. Additionally, in contrast to conventional characterization methods, this technique offers exceptional advantages.

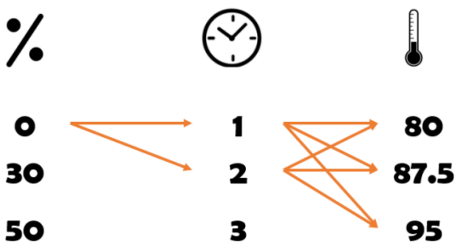

Figure 1. Taguchi method example

Applying many design of experiments (DOE), such as ANOVA, fuzzy logic, complete factorial design, response surface methodology (RSM), and the Taguchi technique, was necessary to optimise the FDM input parameters [13]. Taguchi's design of experiments technique was frequently used to optimise the FDM parameter setting due to its robustness, tolerance, and dimensional control [14]. Ahmad et al. [15] optimised the FDM printing parameters using the Taguchi technique to increase the surface roughness of the produced items. The Taguchi method, on the other hand, is a descriptive survey that pinpoints a process or product in order to improve its usability. The Taguchi technique is used because it is easy to use and a problem-solving method that can help enhance process performance and boost productivity. The strategy is called the test's factorial outline. For every trial, the level combination of information plan variables is chosen using the orthogonal technique [16]. Optimising a process's parameters to achieve maximum efficiency is Taguchi's main objective [17]. The FDM input parameters, including the printing pattern, printing speed, layer thickness, and raster orientation, were optimised using the Taguchi technique. The findings showed that at a 20° displacement angle, layer thickness significantly affected Young's modulus [18,19]. Tensile strength is increased and raw material is conserved when the lowest layer thickness is used [40][20]. Five parameters' effects on the surface integrity of ABS parts made with FDM were examined by Kumar et al. [21,22]. In this work, the ideal set of parameters for the resin casting of composite materials was investigated using Taguchi technique as described above.

2. MATERIALS AND METHODS

The resin that was 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 symbolizes the resin, B the hardener, and C is the accelerator. The proportion of the components according to the manufacturer was not changed. According to the specification given, the proportions are 100 grams of A, 90 grams of B and 2 grams of 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 was 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, a flame retardant was added to the to the mixture. Having known this, the parameters in the process that were changed were three, so we had three factors in our Taguchi matrix. These parameters are as follow:

- 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. Then 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 reason, we used three combinations of temperatures based on the manufacturer sequence of temperatures. One combination would be 7.5 degrees more in each state, one with the manufacturing temperature and the other 15 degrees more, this would help us to see if we go above this temperature (glass transition temperature), will fetch us a better results or make our resin production faster. Considering this, the levels were: (a) 80°C, 120°C, 140°C (b) 87.5°C, 127.5°C, 147.5°C and (c) 95°C, 135°C, 155°C
- Time: This process is very time consuming so we tried to reduce it. Instead of having 3 hours for each phase we tried with one and two hours less, so we had 3+3+3, 2+2+2 and 1+1+1, so we see how this influences the mechanical properties. The less time we consume, the more optimized our process. The levels were (a) 1 hour,1 hour, 1 hour, (b)2 hours, 2 hours, 2 hours and (c)3 hours, 3 hours, 3 hours.
- Flame retardant: To the mixture of resin, we added flame retardant which is a substance that prevents or inhibits the outbreak of fire. We used the following proportions: 0%, 30% and 50%. This proportions were above the

resin we had, not the whole mixture. The goal was to have the greatest proportion of flame retardant without affecting the maximum stress the resin can support. The levels are 0% of flame retardant, 30% of flame retardant and 50% of flame retardant.

After defining the factors and levels, we had 9 factors and 9 levels, which resulted in 27 combinations and therefore 27 experiments. As it would be time consuming and very costly, we applied the Taguchi matrix, which resulted 9 experiments that could guess the best combination of the factors to our purpose. The Taguchi matrix with statistics professional programs (Minitab) was explored. Introducing the data, it was easy to generate the combination below.

	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 2. Experiments conducted generated by the matrix.

Under laboratory conditions, the experimental campaign was carried out with a universal material testing equipment. The loading point was moved at a constant displacement rate of 2 mm/min during the 3-point bending tests. The machine's load cell had a capacity of 2kN. A wire saw machine was used to create the crack in the resin samples, and precracking was also done to sharpen the crack's tip. Figure 4 displays the sample dimensions that are assumed. To obtain a realistic number, the actual dimensions of the incision were measured from the broken specimen in accordance with the standard's instructions.

One of the key characteristics determined by the fracture toughness tests is the material's resistance to fracture and crack propagation. Fatigue pressures, material discontinuities, or manufacturing flaws could all contribute to the crack. It is not always possible to prevent defects from arising during manufacture, therefore it is important to take the fatigue lifetime into account. Numerous crucial details on the mechanism of breaking, such as the type of stresses and the direction of the crack's propagation, may be found by analysing the fracture surface. Figure 5 displays the specimen's breakthrough surface without any additions.

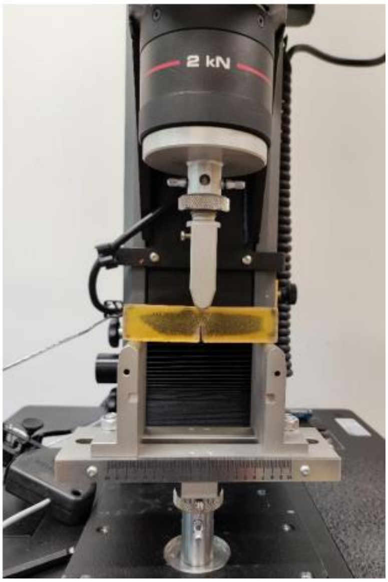

Figure 3. 3-points bending experimental setup.

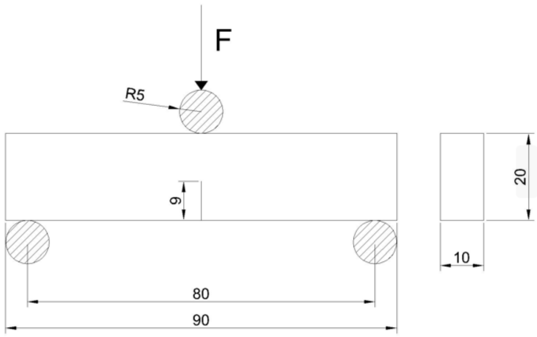

Figure 4. Dimensions of the performed experiment

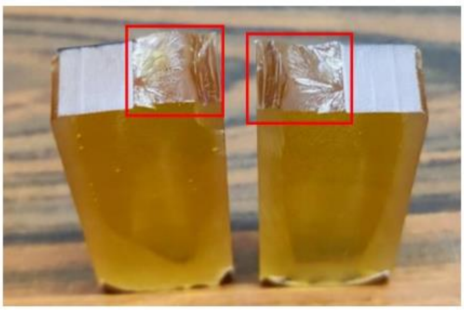

Figure 5. Crack length (sample without flame retardant)

In order to better present the results, a marking system was introduced to facilitate sample identification. Each of the samples has been marked according to the following pattern:

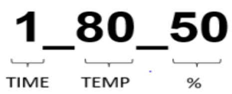

Figure 6. The code of the marked resin specimens

In the case presented on Figure 6 the applied parameters are:

- 1-hour phases,
- 80 degrees of starting temperature
- 50 percentage of the flame retardant

The visualisation of the applied heating cure is on the Figure 7. The slope of the graph is dictated by the need to maintain the maximum heating rate in order to reduce the formation of heat cracks.

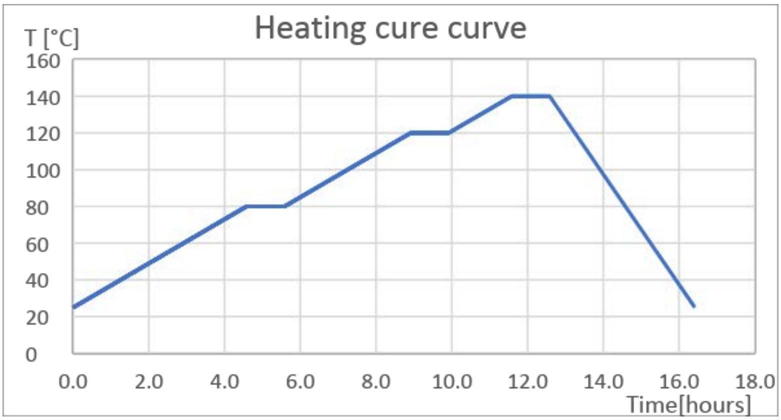

Figure 7. Sample chart of the heating cure curve

3. RESULTS AND DISCUSSION

Figure 8 displays the data collected from the experimental programme. The material's ability to withstand plastic deformation up until fracture was determined by the specimens' mechanical behaviour under load. The stress intensity factor computation and subsequent consideration will make use of linear elastic fracture mechanics. For brittle, homogeneous materials like resin, analysis of the stress intensity factor K allows one to forecast the stress condition close to the notch's tip. Following the test, each specimen was measured in order to determine the sample's geometrical values. The assessment of fracture toughness was done using the greatest force that the testing apparatus could produce.

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

Figure 8. Experimental results

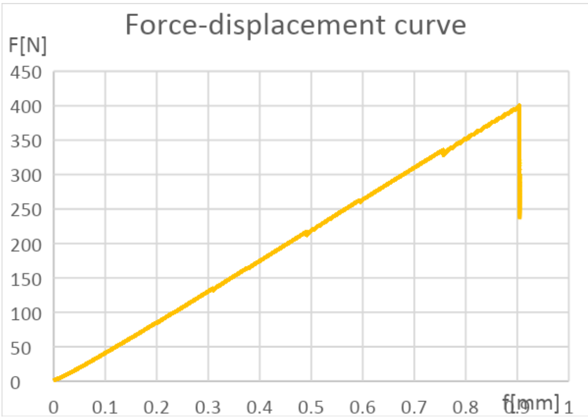

Figure 9. Force-displacement curve of the 3-point bending resin test

The mechanical behaviour of the specimens in the stress state and the response of the testing machine for the applied displacement rate of the loading point are presented on Figure 9.

The stress intensity factor K_I in the loading Mode-I is calculated based on the following equation[23-25]:

$$K_I = \sigma_0 F_I \sqrt{\pi a}$$

$$\sigma_0 = \frac{3PL}{2W^2B}$$

The obtained average values of the resin fracture toughness are presented on Figure 10.

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

Figure 10. Average experimental results

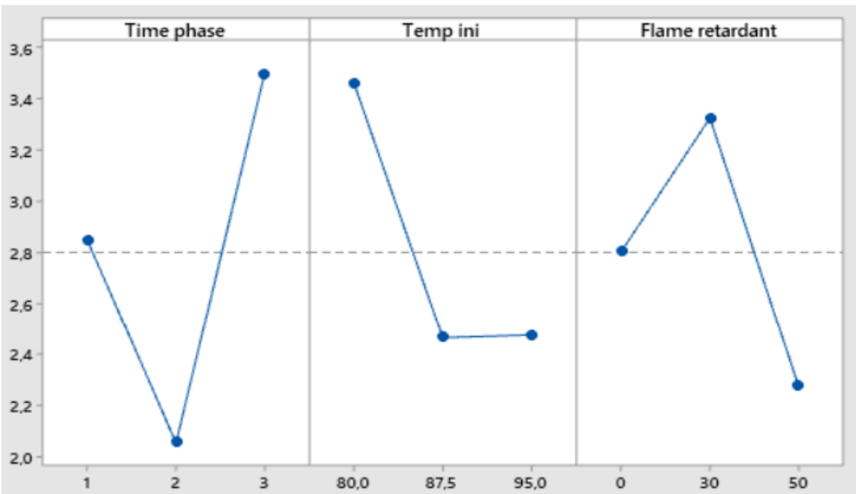

Figure 11. Effects of the parameters on K_I

The experimental campaign's outcomes were utilised to analyse how each element affected the stress intensity factor KI. The ensuing visuals display the outcomes. The mean value for the whole set of data is shown by the dashed line.

- Time of each phase

From the perspective of the manufacturer, process time is an important consideration. A decrease in time results in a decrease in the company's expenses. The goal is to choose the solution that best fits the needs of the product. The one- or three-hour time cure in this instance should be taken into account. The producer's suggested time represents the factor's optimal value. Reducing the phase's duration may be beneficial if the fracture toughness is at the safety level.

- Temperature

The fracture toughness characteristic decreases when the process temperature rises. Increased temperatures result in energy waste and needless expenses. The value that the datasheet recommends is the best choice.

- Flame retardant

The resin microstructure is affected differently by the addition of flame retardants, such as aluminium trihydroxide, depending on the additive's concentration, particle shape, and surface preparation. According to the data, 30% of the particles are the optimum option for the resin's characteristics.

The conducted research allows to verify the parameters recommended from the resin manufacturer and the influence of the various factors on the mechanical properties such as fracture toughness. The best results were obtained from the temperature sequences 80°C, 120°C, 140°C, 3 hours of the temperature standstill and 30 percent of the flame retardant.

4. CONCLUSIONS

The primary goal of designing experiment methods, such the Taguchi method, is to optimise the parameters of the product through the utilisation of multiple components in the trials. These methods help cut down on the quantity of experiments conducted and conserve time, energy, and money. This approach's primary drawback is how mistakes affect the way the experiments turn out. More accuracy and preservation of condition should be used in the investigation. Errors made during a campaign have a greater influence on the outcome and are more difficult to identify.

REFERENCES

[1] Mouritz AP. *Introduction to aerospace materials*. Elsevier; 2012.

[2] Sojka SA, Moniz WB. *The curing of an epoxy resin as followed by carbon-13 NMR spectroscopy*. J Appl Polym Sci 1976;20:1977–82.

[3] Cañavate J, Colom X, Pages P, Carrasco F. *Study of the curing process of an epoxy resin by FTIR spectroscopy*. Polym Plast Technol Eng 2000;39:937–43.

[4] González MG, Cabanelas JC, Baselga J. *Applications of FTIR on epoxy resins-identification, monitoring the curing process, phase separation and water uptake*. Infrared Spectroscopy-Materials Science, Engineering and Technology 2012;2:261–84.

[5] Massingill Jr JL, Pabon Jr RA. *Modified advanced epoxy resins* 1989.

[6] May C. *Epoxy resins: chemistry and technology*. Routledge; 2018.

[7] Wu F, Zhou X, Yu X. *Reaction mechanism, cure behavior and properties of a multifunctional epoxy resin, TGDDM, with latent curing agent dicyandiamide*. RSC Adv 2018;8:8248–58.

[8] Erdmann M, Trappe V, Sturm H, Braun U, Duemichen E. *Cure conversion of structural epoxies by cure state analysis and in situ cure kinetics using nondestructive NIR spectroscopy*. Thermochim Acta 2017;650:8–17.

[9] Mphahlele K, Ray SS, Kolesnikov A. *Cure kinetics, morphology development, and rheology of a high-performance carbon-fiber-reinforced epoxy composite*. Compos B Eng 2019;176:107300.

[10] Rogers MG. *The structure of epoxy resins using NMR and GPC techniques*. J Appl Polym Sci 1972;16:1953–8.

[11] Merad L, Cochez M, Margueron S, Jauchem F, Ferriol M, Benyoucef B, et al. *In-situ monitoring of the curing of epoxy resins by Raman spectroscopy*. Polym Test 2009;28:42–5.

[12] Wang LW, Fernando GF. *Cure monitoring of epoxy resin by simultaneous DSC/FTIR*. Adv Mat Res 2014;881:905–8.

[13] Uttarwar PB, Raini SK, Malwad DS. *Optimization of process parameter on Surface Roughness (Ra) and Wall Thickness on SPIF using Taguchi method*. International Research Journal of Engineering and Technology 2015;2:781–4.

[14] Naiju CD, Anil PM. *Influence of operating parameters on the reciprocating sliding wear of direct metal deposition (DMD) components using Taguchi method*. Procedia Eng 2017;174:1016–27.

[15] Lee BH, Abdullah J, Khan ZA. *Optimization of rapid prototyping parameters for production of flexible ABS object*. J Mater Process Technol 2005;169:54–61.

[16] Uddin MS, Sidek MFR, Faizal MA, *Ghomashchi R, Pramanik A. Evaluating mechanical properties and failure mechanisms of fused deposition modeling acrylonitrile butadiene styrene parts*. J Manuf Sci Eng 2017;139:081018.

[17] Hamza I, Abdellah EG, Mohamed O. *Experimental optimization of fused deposition modeling process parameters: a Taguchi process approach for dimension and tolerance control*. Proceedings of the international conference on industrial engineering and operations management, 2018, p. 2992–3.

[18] Ahmad MN, Ab Rahman MH, Maidin NA, Osman MH, Wahid MK, Mohamed Saiful Firdaus H, et al. *Optimization on Surface Roughness of Fused Deposition Modelling (FDM) 3D printed parts using taguchi approach. Intelligent Manufacturing and Mechatronics: Proceedings of the 2nd Symposium on Intelligent Manufacturing and Mechatronics–SympoSIMM 2019, 8 July 2019, Melaka, Malaysia*, Springer; 2020, p. 230–43.

[19] Ahmad MN, Ab Rahman MH, Maidin NA, Osman MH, Wahid MK, Mohamed Saiful Firdaus H, et al. *Optimization on Surface Roughness of Fused Deposition Modelling (FDM) 3D printed parts using taguchi approach. Intelligent Manufacturing and Mechatronics: Proceedings of the 2nd Symposium on Intelligent Manufacturing and Mechatronics–SympoSIMM 2019, 8 July 2019, Melaka, Malaysia*, Springer; 2020, p. 230–43.

[20] Qattawi A. *Investigating the effect of fused deposition modeling processing parameters using Taguchi design of experiment method*. J Manuf Process 2018;36:164–74.

[21] Onwubolu GC, Rayegani F. *Characterization and optimization of mechanical properties of ABS parts manufactured by the fused deposition modelling process*. International Journal of Manufacturing Engineering 2014;2014:1–13.

[22] Uddin MS, Sidek MFR, Faizal MA, Ghomashchi R, Pramanik A. *Evaluating mechanical properties and failure mechanisms of fused deposition modeling acrylonitrile butadiene styrene parts*. J Manuf Sci Eng 2017;139:081018.
[23] Fett, T.; Gerteisen, G.; Hahnenberger, S.; Martin, G.; Munz, D. *Fracture tests for ceramics under mode-I, mode-II and mixed-mode loading*. J. Eur. Ceram. Soc. 1995, 15, 307–312.
[24] Fett, T. *Stress intensity factors for edge crack subjected to mixed mode four-point bending*. Theor. Appl. Fract. Mech. 1991, 15, 99–104.
[25] Araki, W.; Nemoto, K.; Adachi, T.; Yamaji, A. *Fracture toughness for mixed mode I/II of epoxy resin*. Acta Mater. 2005, 53, 869–875.

Corresponding author:
grzegorz.lesiuk@pwr.edu.pl