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EXPERIMENTAL DESIGNS USED IN THE FABRICATION OF POLYESTER-MWCNT NANOCOMPOSITES

Kashif Ullah Khan[1,2], Andrea Ádámné Major [2]*

[1] Doctoral School of Materials Sciences and Technologies, Óbuda University, Hungary, https://orcid.org/0009-0003-3345-2484
[2] Department of Innovative Vehicles and Materials, GAMF Faculty of Engineering and Computer Science, John von Neumann University, Hungary, https://orcid.org/0009-0009-8546-718X

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Abstract

This article highlights the various design of experiment approaches employed for developing nanocomposites from polyester/MWCNT. To overcome the complexity between different factors like MWCNT content, processing conditions, and properties of nanocomposites, the different approaches such as fractional factorial design (FFD), Taguchi, and response surface methodology (RSM) were used. Though tremendous progress has been observed in the field of nanocomposites, there is still a dearth of studies that compare the efficiency of RSM, FFD, and Taguchi technique for polyester MWCNT nanocomposites. This review addresses that gap directly by focusing on polyester/MWCNT systems specifically and by following the progression an experimenter would take, from screening the relevant factors with FFD, to optimizing them for robustness with Taguchi design, to building a predictive model with RSM. The RSM was used to obtain the predictive mathematical models. However, the FFD was used to evaluate the significant factors and Taguchi for processing parameters. It was found out that dispersion of MWCNT and temperature during processing are the most influential factors. Overall, FFD proved most effective for identifying the significant variables with a minimum number of experiments, Taguchi design enabled robust optimization of processing parameters under variability, and RSM gave the most detailed modeling of nonlinear interactions with the strongest predictive capability.

1 Introduction

The polymer nanocomposites modified by multi-walled carbon nanotubes (MWCNT) have recently become one of the promising advanced materials characterized by improved mechanical, thermal, and electrical characteristics [1], [2]. Among various types of polymers, thermoplastics, including polyethylene terephthalate (PET), polybutylene terephthalate (PBT), polylactic acid (PLA), and polybutylene succinate (PBS), receive much interest because of their high practical significance, good processability [3], [4], [5], [6]. PET and PBT are considered as engineering plastics that find extensive application in automotive engineering, electronics, and packaging industry due to excellent dimensional stability and high mechanical resistance, whereas PLA and PBS belong to the group of

* Corresponding author.
E-mail address: major.andrea@nje.hu

biodegradable polyesters that are widely applied in the development of sustainable materials and biomedical devices [7], [8], [9].

Modification of thermoplastic polyester by MWCNT can lead to an essential increase in mechanical and thermal properties, mainly due to the creation of a continuous nanotube network and good interfacial interactions [10], [11]. However, the degree of modification highly depends on several interconnected parameters, including the concentration and dispersion state of nanotubes, their functionalization and crystallization behavior, as well as processing parameters like melt temperature, screw speed, residence time, and cooling rate [12], [13]. In semicrystalline polymers MWCNT shows a nucleating effect that significantly enhance the mechanical and thermal properties of nanocomposites. Such phenomena were observed in polyester based systems wherein MWCNT contributed to improved crystallinity and phase transformation [14]. The addition of MWCNT in PLA matrix solved the inherited brittleness and enhanced the toughness of nanocomposites. However, high amount of MWCNT content decreased the mechanical properties including filler agglomeration [15], [16]. Thus, to achieve a satisfactory performance in terms of mechanical properties and multifunctionality, optimal levels of several factors should be determined.

The traditional approaches have limitations to explain all possible interaction with one factor variations. Therefore, more sophisticated approaches, such as response surface methodology (RSM) [17], Taguchi Design [18], and fractional factorial design (FFD) [19], are highly required for the fabrication and optimization of polyester MWCNT nanocomposites. One of the approaches that allows modeling the interactions among various factors in PET, PBT, PLA, and PBS is RSM. This technique can be used to establish models of responses, such as tensile strength, electrical conductivity, crystallinity, and thermal stability, and determine optimal levels of the factors with minimum number of experiments. The RSM approach was used widely to design melt processing parameters, filler content, and dispersion of the nanocomposite [20]. Factorial designs are very helpful in the preliminary screening of numerous factors. In the case of nanocomposites, factorial designs have been employed successfully to find critical factors for the improvement of mechanical properties and optimization of processing conditions [21]. However, Taguchi design represents an optimization technique based on orthogonal array and signal-to-noise ratio that makes it possible to determine optimal experimental conditions with account for variations (Table 1). This approach has been employed for optimization of processing conditions in the case of polymer nanocomposites with consideration of improved mechanical and electrical performance [8].

While there is less literature concerning design of experiments (RSM/Taguchi/FFD) involving PET, PBT, PBS with MWCNT than on PLA, the DOE approaches validated for PLA are often assumed to transfer to these other polyesters because of their broadly similar melt processing and crystallization behavior, though this assumption has rarely been tested directly. Even though the mentioned approaches were used independently, it is important to systematically analyze and integrate the RSM, FFD, and Taguchi methodologies for optimization purposes.

2 Types of experimental designs

2.1 Fractional Factorial Design (FFD)

The FFD [22] used 2^(5-1) design to investigate the effect of five factors at two levels (Table 1). FFD is identify the significant factors (equation 1). Although FFD efficiently reduces the number of experiments required for screening, it cannot capture higher-order interactions between factors, which limits its applicability in strongly nonlinear systems.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i<j} \beta_{ij} X_i X_j \quad (1)$$

Where:
Y= Responses factors
β_i = main effect coefficient *β_{ij} = interaction effects*
X_i = coded variables

2.2 Taguchi Method

In Taguchi method [23] an L9 orthogonal array method was adopted in optimizing parameters at each three levels (Table 1). Taguchi determine the robust processing parameters. Equation 2 was employed to determine the larger to better properties such as tensile strength, and conductivity. A drawback of this approach is that it generally assumes the control factors act independently, so interaction effects between them can be missed unless they are built into the array beforehand.

$$S/N = -10\log_{10}\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right) \quad (2)$$

Where:
S/N= signal to noise ratio
y_i= Responses values
n = number of experiments

Table 1. Factors and levels for experimental designs

Control factors	Symbol	Experimental factor levels	Nature
MWCNT (wt.%)	A	0.5, 1.5, 3.0	Nanotube concentration
Temperature (°C)	B	180–290 (depending on polymer)	Melt/extrusion temperature
Screw speed (rpm)	C	50, 100, 150	Shear intensity
Mixing time (min.)	D	5, 10, 15	Residence time
Cooling rate (°C/min.)	E	5, 10, 20	Crystallization control

2.3 Response Surface Methodology (RSM)

Central Composite Design (CCD) was adopted in optimizing significant parameters obtained from FFD (Table 1). RSM refine interactions and obtain global optimizations [24]. Second order polynomial model shows in equation 3. This modeling power comes at a cost, however, since CCD generally needs more experimental runs than FFD or Taguchi arrays, and the fitted polynomial can lose accuracy once the true response departs substantially from the region covered by the design.

$$Y = \beta_0 + \sum_{i=1}^{k}\beta_i X_i + \sum_{i=1}^{k}\beta_{ii} X_i^2 + \sum_{i<j}\beta_{ij} X_i X_j + \varepsilon \quad (3)$$

Where:
Y= Response
β_0 = intercept *β_i = linear effect*
β_{ii} = quadratic effect *β_{ij} = interaction effects*
X_i = Independent variables *ε = experimental error*

3 Experimental designs used for nanocomposites fabrications

3.1 FFD

FFD has been extensively used to determine the most significant variables in the polymer/MWCNT nanocomposites, especially if there are many parameters. In this case, Kuo et al. [25] were synthesized MWCNT using a chemical vapor deposition floating feed method in a vertical reactor. Effects of the preparation variables on the average diameter of carbon nanotubes were examined using the FFD methodology. The results indicated that the diameter of carbon nanotubes generally increased with increasing reaction temperature and methane flow rate. Furthermore, research was performed by Aoki et al. [26], they came up with a full theoretical basis that could be applied in analyzing and creating fractional factorial designs. The authors explained that FFD was able to capture dominant main effects as well as low order interaction effects through much fewer experiments. According to the researcher, FFD was very applicable especially where there were several influencing factors affecting the outcome of the product. For example, in the case of

polymer/multifunctional carbon nanotubes composite, which involves several parameters such as processing temperature, nanotube content, screw speed, residence time, and cooling processes (Table 2).

Moreover, Tang et al. [21] carried out an analysis on advanced uniform FFD method that possess features of classical factorial designs and space filling properties too. The objective of this study was to enhance experimental efficiency with respect to systems where highly nonlinear responses with multiple interacting factors exist. According to Tang et al., uniform FFD methods is especially useful for advanced materials optimization processes such as polymer nanocomposite optimization processes and multi-functional material optimization. This study composite systems. Furthermore, a validation study was conducted by Toapanta et al. [27], they tested the efficiency of FFD techniques as a preliminary stage in developing polymer composite materials. Based on their findings, FFD considered as a highly efficient and reliable technique for the determination of significant factors before applying advanced models such as RSM to further optimization and accurate characterization of material properties. Thus, the discussed examples show the importance of applying factorial design at the stage of screening parameters before optimization.

Table 2. FFD method used in the fabrication of different nanocomposites.

Polymer system	Output factor	Improvement	Dominant factors	Reference
MWCNT	Diameter	increases	Temperature	Kuo et al., 2005, [25]
Polymer/MWCNT	Model accuracy	Good	Interaction effects	Aoki et al., 2012, [26]
Polymer nanocomposites	Model accuracy	Significant	Interaction effects	Tang et al., 2012, [28]
Polymer systems	Model accuracy	$R^2 \approx 0.95$	Design efficiency	Toapanta et al., 2024, [27]

3.2 Taguchi Method

Taguchi design has become a popular methodology used for optimizing nanocomposites reinforced with polymer MWCNT because of its high efficiency and capability to minimize efforts needed for experiments by accounting for variations in the process. In this case, Suherman et al. [29] used Taguchi methodology to enhance electrical conductivity of polymer MWCNT nanocomposites. To obtain the good conductive network, they found that nanotube content, mixing procedure, and processing temperature are the responsible factors. Thus, based on signal to noise ratio analysis, they revealed the optimal settings that resulted in improved electrical conductivity and minimized experimental variability. The findings of the Taguchi method revealed a high dependence of electrical conductivity on MWCNT loading and process temperature. The electrical conductivity was found to increase from the region of insulator (~10^{-12} S/cm) to conductive (~10^{-2} S/cm), with increase in the amount of MWCNT loading from 0.5 wt% to 3 wt%, with a percolation limit close to 1-1.5 wt%. The Mashhadzadeh et al. [30] studied the effect of processing conditions on the mechanical performance of polyester based nanocomposites. Using the Taguchi design, they found that the processing parameters influenced the tensile and flexural properties of polyester MWCNT materials. In the results, they showed that dispersion and interfacial bonding are the main factors determining the mechanical performance of nanocomposites. Therefore, the methodology helped to determine important parameters (Table 3).

As for biodegradable polyesters, Gowtham Raj et al. [31] applied the Taguchi optimization method for vinyl ester-based materials. According to the findings, the process parameters such as mixing temperature and time have a significant effect on the dispersion of nanofillers. This improves the mechanical properties of the material and reduces agglomeration, which is among the major issues in manufacturing nanocomposites. Longer time during the mixing process increases uniform distribution of MWCNT. The use of polymer nanocomposites as materials for various engineering applications was discussed by Chan-Chan et al. [32], researcher utilized Taguchi methodology to explore the effect of different process parameters on performance characteristics. They concluded

that the use of Taguchi method allowed them to identify appropriate values of temperature, pressure, and filler content that would help to improve material performance and stability.

Furthermore, the use of Taguchi design was discussed in electrospun biopolymer nanocomposites by Kalluri et al. [33], applying Taguchi method, they successfully attained the improved mechanical and functional performance of fibers through proper electrospinning and processing parameters. It becomes clear that the technique is highly effective because it allows researchers to analyze a variety of parameters. Moreover, the method is especially useful when dealing with such nanocomposites as PET, PBT, PLA, and PBS.

Table 3. Taguchi method used in the fabrication of different nanocomposites.

Polymer system	Output factor	Improvement	Dominant factors	Reference
Polymer/MWCNT	Conductivity	~10^{10}× increase	MWCNT loading	Suherman et al., 2011, [29]
Polyester nanocomposites	Tensile strength	~25–40%	Filler content	Mashhadzadeh et al., 2017, [30]
PLA-based systems	Dispersion	~30–50%	Mixing time	Gowtham Raj et al., 2023, [31]
Polymer nanocomposites	Thermal stability	+10–20 °C	Temperature	Chan-Chan et al., 2020,[32]
Biopolymer systems	Fiber uniformity	~30–50%	Voltage	Kalluri & Duan, 2022, [33]

3.3 RSM

Various studies explored the effectiveness of RSM in the optimization of processing parameters and improvement of the properties of MWCNT reinforced polymer systems. RSM significance was highlighted by Raza et al. [34], they found that the prediction errors using the RSM approach to quadratic regression were always within the range of 0.90 to 0.99 for mechanical, thermal, and electrical properties of polymer nanocomposites. In this same review, the authors reported that RSM reduced the number of experiments by roughly 40–70% relative to one variable at a time experimentation, and that combining RSM with artificial intelligence improved prediction accuracy by roughly 15–25% relative to purely statistical approaches for nonlinear polymer systems. Moreover, Alateyah et al. [35] used RSM in the optimization of processing parameters in polyester based composites. Although this study concerns machining rather than fabrication, it is included here because it demonstrates RSM's capacity to model multi-factor interactions in a polyester-based system, a capability that is directly transferable to the fabrication parameters discussed in this review. The study demonstrated that the temperature, feed rate, and machining conditions significantly influence material properties. The study suggests that RSM can be considered an efficient and reliable technique for modeling the relationship between processing variables and mechanical properties (Table 4).

Furthermore, Madaeni at al. [36] studied the impacts of the amount of polymer, time of evaporation, and concentration of PVP added on the properties of membrane morphology and filtration. While this application is membrane fabrication rather than a polyester/MWCNT nanocomposite, it is discussed here because it shows RSM's strength in modeling interaction effects between formulation variables, the same strength that makes RSM attractive for polyester/MWCNT systems. In the optimized membrane, they noted that the water flux was significantly higher, with more efficient salt rejection with less structural faults. The regression models that were generated have shown low prediction errors and good statistical adequacy, indicating high significance of the models. It has been proven that the interaction effects of polymer concentration and evaporation time greatly impact on the pore formation and permeability of the membrane. In addition, Toapanta et al. [27] carried out an extensive comparative analysis of various experimental design techniques, including RSM, for polymer systems. Their investigation confirmed the effectiveness and predictive capacity of RSM in modeling complex material behavior and optimization of various responses. Furthermore, they highlighted the importance of RSM over to other conventional experimental approaches in case of the reduction of experiments while maintaining accuracy.

Therefore, these studies collectively show the suitability of RSM in designing and optimizing MWCNT reinforced polymer-based materials. The capability of modeling non-linear interaction effects provides additional advantages of RSM in complex systems.

Table 4. RSM method used in the fabrication of different nanocomposites.

Polymer system	Output factor	Improvement	Dominant factors	Reference
Polymer nanocomposites	Prediction accuracy	15–25%	Quadratic regression	Raza et al., 2025, [34]
Polyester composites	Strength	+20–35%	Temperature	Alateyah et al., 2022, [35]
ABS/PVP	Pore formation	Significant	Polymer concentration and evaporation time	Madaeni et al., 2011, [36]
Polymer systems	Model accuracy	~10^{10}× increase	Interaction effects	Toapanta et al., 2024, [27]

3.4 Summary

Placed side by side, the three techniques each serve their own purposes instead of being substitutes for the same task. FFD is the method to use when one needs to reduce the number of candidate variables from a large list at minimal expense. Taguchi is the method to use when one needs to fix processing parameters for practical conditions, and RSM is the method to use when one wants to predict the response behavior over the design region including the interaction effects. Table 5 shows this comparison.

Table 5. Comparison of FFD, Taguchi, and RSM for polyester/MWCNT nanocomposite development.

Method	Primary purpose	Typical run count	Main limitation
FFD	Screening a large set of factors down to the significant few	Low (fractionated design)	Cannot resolve higher-order interactions
Taguchi	Optimizing processing parameters for robustness to noise	Low to moderate (orthogonal array, e.g. L9)	Assumes factors act independently and interactions can be missed
RSM	Modeling nonlinear responses and interaction effects for prediction	Higher (central composite or similar design)	Requires more runs and accuracy drop outside the fitted region

4 Conclusions

As can be seen from the literature review, such methodology for the optimization and development of nanocomposites as statistical and design of experiments techniques, including FFD, Taguchi Design, and RSM, proved to be crucial in this context. Various polymers were considered in the studies, such as PET, PBT, PLA, PBS, and others based on polymer matrix. Using the discussed methodologies, the important processing and formulation variables responsible for dispersion quality, crystallization, and final properties were identified successfully.

However, FFD was used as an efficient technique for screening of the initial large number of variables down to the most important factors with a minimum number of experiments required. Such a screening is especially useful when dealing with nanocomposites because of the great number of interacting parameters to take into consideration. Moreover, the use of Taguchi design was found to be quite effective, allowing researchers to perform parameter optimization considering the effect of variability in processing condition while requiring a small number of experiments. In turn, RSM enabled more precise modeling of variable interaction and nonlinear response relationship that could lead to optimization of several material characteristics at once. Therefore, the discussed studies can illustrate the progression in the experimental design process of nanocomposites, starting from the screening using FFD, moving to parameter optimization using Taguchi Design, and ending with

material response modeling and multivariable optimization in RSM. For future research activity, the number of studies conducted using experimental design (RSM, Taguchi, FFD) approaches for PET, PBT, and PBS with MWCNT nanocomposites is relatively low in comparison with those conducted using PLA.

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