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EXPERIMENTAL STUDY ON MECHANICAL PROPERTIES OF POLYAMIDE-6/ MULTI-WALLED CARBON NANOTUBES NANOCOMPOSITE FOAMS

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ABSTRACT

In this paper, first polyamide 6 (Nylon 6) melt is compounded with multi-walled carbon nanotubes (MWCNTs) using a twin-screw extruder with variant weight percentages of MWCNTs (i.e.0, 0.5, 1 and 1.5 wt%). Then, the prepared nanocomposites are foamed using Azodicarbonamide as a blowing agent in an injection molding machine. The foamed nanocomposite specimens are produced according to a L16 orthogonal array of Taguchi Design of Experiments (DOE). The influence of variable parameters, including the amount of MWCNTs, holding pressure and holding pressure time (all in four levels)is investigated on mechanical properties of nanocomposite samples. Tensile strength, hardness and impact strength of samples are investigated as mechanical properties of nanocomposite foam specimens. According to the results of the tests, the amount of MWCNTs is the most effective parameter on tensile strength, hardness and impact strength of the foamed nanocomposite samples as adding 1 wt% of MWCNTs causes 147% improvement in specific tensile strength and 1.5 wt% of MWCNTs causes about 17% enhancement in Rockwell hardness of samples. Also the specific impact strengths of nanocomposite foam samples decreased by addition of MWCNTs.

Keywords: Nanocomposite; foam; PA-6; MWCNT; Mechanical properties

1. INTRODUCTION

Polymer nanocomposite foams have increasingly received attention in both scientific and industrial communities; because the combination of functional nanofillers and foaming technology has the potential to generate a new class of materials that are light, strong, and multifunctional [1, 2]. Polymer nanocomposite foams are important materials in a variety of applications due to their inherent advantages, such as low density, high specific strength, light weight, materials savings, low thermal conductivity and good sound isolation [3, 4].

In the other hand, polymer nanocomposites are an important class of materials, which are an alternative to the conventionally filled polymers. These are kinds of the materials in which nanosized inorganic fillers, typically 1–100 nm in at least one dimension, are individually dispersed in a polymer matrix. Because of the nanometer sizes of the fillers, nanocomposites exhibit markedly improved properties in comparison with pure polymers or their conventional composites. For instance it includes increase of the modulus and strength, noticeable barrier properties, improve of solvent, heat resistance, and decreasing of flammability [1, 5, 6]. Using nanocomposites in foam formation enhances their property profiles and enabling a broader range of uses, from conventional to advanced applications [1].

Microcellular foams, which are characterized by cell size between 1 to 100 microns, provid improved mechanical and thermal properties such as high strength to weight ratio, and enhanced toughness and fatigue life of parts [7]. According to the size of the foam cells, polymer foams can be classified as macrocellular (>100 μm), microcellular (1–100 μm), ultramicrocellular (0.1–1 μm) and nanocellular (0.1–100 nm). The synthesized nanocomposites can be used to produce nanocomposite foams. For large-scale production, the direct utilization of foaming (blowing) agents is the most commonly used method. Foaming agents are substances that form the gaseous phase in the foams. Two types of foaming agents are often used: physical and chemical blowing agents. Chemical foaming agents are usually reactive species that produce gases in the foaming process, while physical foaming agents are substances that gasify under foaming conditions [1, 8].

Polyamide 6 (nylon 6) is an important engineering semi crystalline thermoplastic with a wide range of engineering applications, which is often reinforced with fibers in practice. Nylon 6 finds application in a broad range of products requiring materials of high strength [9, 10]. It is widely used for gears, fittings, and bearings, in automotive industry for under-the-hood parts, and as a material for power tools housings. Nylon 6 is used as thread in bristles for toothbrushes, surgical sutures, and strings for acoustic and classical musical instruments, including guitars, sitars, violins, violas, and cellos [11, 12].

Since their discovery in the early 1990s, both multi-walled (MWNTs) and single-walled carbon nanotubes (SWNTs) have received considerable attention due to their unique and remarkable mechanical, electrical and thermal properties [13, 14]. In summary, three properties of MWCNTs are specifically interesting for the industry: the electrical conductivity (as conductive as copper), their mechanical strength (up to 15 to 20 times stronger than steel and 5 times lighter) and their thermal conductivity (same as that of diamond and more than five times that of copper). A combination of these impressive properties enables a new variety of useful and beneficial applications. Therefore, carbon nanotubes (CNTs) have been considered for a wide range of potential applications, especially as fillers in polymer composites [15].

Zeng et al. synthesized poly (methyl methacrylate) (PMMA) and multi-walled carbon nanotubes (MWCNTs) and foamed the nanocomposites by supercritical carbon dioxide. They studied morphology and tensile properties of both solid and foamed nanocomposites. Nanocomposite foam with concurrent increases in tensile strength (~40%), tensile modulus (~60%) and strain at break (~70%) was successfully prepared using 0.5% of functionalized CNTs that were well dispersed [2].

Chen et al. used multi-walled carbon nanotubes (MWNTs) with controlled aspect ratio to alter the foam morphology in MWNT/Poly(methyl methacrylate) (PMMA) nanocomposite foams produced by a supercritical carbon dioxide (CO_2) foaming process. They found that adding 1 wt% of MWNTcauses 82% increase in Young's modulus and 104% increase in collapse strength of polymer foams [16].

Park et al. prepared ethylene vinyl acetate (EVA)-MWCNT nanocomposite foams by chemical foaming and observed significant increase in tensile strength and modulus accompanied by decrease in elongation [17].

V.Dolama et al. produced pure and nano-reinforced polyurethane foams by an in-situ polymerization technique. They used carbon nanotubes (single-walled carbon nanotubes and multi-walled carbon nanotubes) and carbon nanofibers as reinforcing particles. They studied the alterations of morphology and mechanical properties of the nano-reinforced polyurethane foams. Compression tests revealed an improvement in both compressive Young's modulus and compressive strength, and scanning electron microscope (SEM) observations showed a decrease in the average cell size of the foam. They reported 97% improvement in compressive young's modulus in foam nanocomposites containing 0.5 wt% MWNTs [18].

In this study, PA-6 has been melt compounded with multi-walled carbon nanotubes by a twin-screw extruder and the obtained nanocomposite has been foamed by chemical method using Azodicarbonamide as a blowing agent in an injection molding machine. The effect of variable parameters on mechanical properties of nanocomposite foamed specimens has been investigated using Taguchi Design of Experiments. Considered variable parameters of this study include: the amount of MWCNT, holding pressure and holding pressure time. Tensile strength and Rockwell hardness of the nanocomposite foamed specimens have been investigated and finally the toughness of specimens has been studied using impact strength test.

2. Experimental details

2.1. Materials

In this study commercially PA-6 (with Trade name ofTecomide NB40 NL E, purchased from Eurotec, Turkey) with density of 1.13 g/cm^3, suitable for extrusion and molding process, has been used as the polymeric matrix of present study. Industrial grade of MWCNTs with purity of 90% supplied by US Research Nanomaterials, Inc. is used in exprimentations.Inner diameters, outer diameters and length of nanotubes are 5-10 nm, 10-30 nm, and 10-30 μm respectively. Azodicarbonamideis also used as the blowing agent in chemical foaming process of PA-6/MWCNT nanocomposites.

2.2. Equipment

A ZSK-25 (Coperion Werner &Pfleiderer, Germany) twin-screw extruder with 10 kg/h extruding capacity has been used for melt compounding of the materials and an NBM HXF-128 injection molding machine with L/D=21.1 and D=37mm of screw, has been used for injection molding of specimens.A Gotech-AI-7000M tensile test machine used to calculate tensile strength of nanocomposite foam samples with 10 mm/min speed of test. An Indentec universal hardness test machine

(Zwick/Roell, England) used for Rockwell hardness tests and acharpy impact test machine with pendulum mass of 2.036 kg and arm length of 39.48 cm has been used to determine the impact strength of samples.

2.3. Design of Experiments (DOE)

The Design of Experiments (DOE) is an important statistical method that is widely used to enhance the quality of the products. Usingthismethod, we canrecognizeresources thatmay affectthequality of the products and selectthebestanswerby changing thesizeof the factorsin a controlled environment perusing the different responses. Some of the variable factors such as$x_0, x_1, \ldots\ldots x_p$ are controllable and the others $z_0, z_1, \ldots\ldots z_q$ are uncontrollable (Fig. 1) [19, 20].

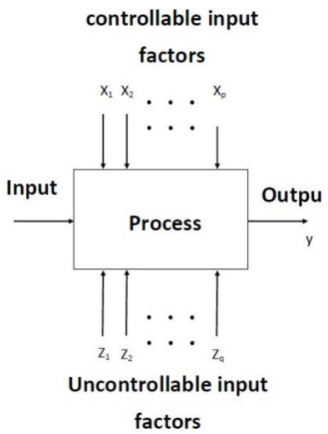


Fig.1. Controllable and uncontrollable factors

Taguchi method can reduce the number, time and cost of the experiments several times, with high confidence in the systems which study of the factors with different levels is needed to determine the optimal conditions. Also this method can predict the system's optimal responseeven whenit is not been examined inthetests. Taguchi method introduces a loss function that is presented as signal to noise ratio (S/N ratio). Depended on the test, each forms of Equation (1-1, 1-2 and to 1-3) can be used to determine S/N ratio [20, 21].

$S/N=10\log[\frac{\bar{Y}^2}{S^2}]$	Nominal is best	(1-1)
$S/N=-10\log[\frac{1}{n}\sum_{i=1}^{n}\frac{1}{{y_i}^2}]$	Larger is better	(1-2)
$S/N=-10\log[\frac{1}{n}\sum_{i=1}^{n}{y_i}^2]$	Smaller is better	(1-3)

In each problem, regardless to kind of it, when the S/N is the largest, the received response is optimal.At the present research, Minitab software has been used for statistical analysis ofTaguchi method. Since in the present study the largest amount of mechanical properties is desirable, "larger is better" method (Eq.1-2) is used in Taguchi approach to analyzing data.

2.4. Preparation of specimens

Before melt compounding, PA-6 is dried using a laboratory oven at 120 °C for 2 hours in order to eliminate the humidity of polymer. Then, PA-6 and MWCNTs are extruded in a twin-screw extruder with different weight percentages of MWCNT at melt temperature of 240 °C and screw speed of 250 rpm to produce the nanocomposite granules. After that, the obtained nanocomposite granules are dried in the hopper of injection molding machine at 80 °C for 20 hours. In order to provide better foaming conditions, 2 wt% of Azodicarbonamide and 1 wt% of paraffin oil are added to nanocomposite granules as blowing agent and softening materials respectively. Table 1 shows the weight percentages of the materials used in this research.

According to processing parameters and their levels (shown in Table 2), the L16 orthogonal array of Taguchi method is selected to design experiments asTable 3 [20]. The constant parameters of injection molding process are also shown in Table 4.

The combined materials are injected to a plate shape mold cavity using the injection molding machine. The mold of injection machine and samples of the injected plates are shown in Fig. 2 and Fig. 3; respectively. The mold has a length of 17.5 centimeters, width of 8 centimeters, and thickness of 3.2 millimeters. Then, specimens are cut from the injected plates using a NCC9012 CNC laser cutting machine based on the ASTM:D638-10 and ASTM:D6110 [19] standard of tensile and impact test; respectively.

Table 1: Weight percentages of materials used in present study

Level / Material	1	2	3	4
MWCNT (wt%)	0	0.5	1	1.5
Azodicarbonamide (wt%)	2	2	2	2
Paraffin (wt%)	1	1	1	1
PA-6 (wt%)	97	96.5	96	95.5

Table 2: Processing parameters and their levels

Level / Parameter	1	2	3	4
MWCNT (wt%)	0	0.5	1	1.5
Holding pressure time (s)	1	2	3	4
Holding pressure (MPa)	80	100	120	140

Table 3: L16 orthogonal array of Taguchi Design of Experiments

Parameter / Trial	MWCNT (wt%)	Holding pressure time (s)	Holding pressure (MPa)
1	0	1	80
2	0	2	100
3	0	3	120
4	0	4	140
5	0.5	1	100
6	0.5	2	80
7	0.5	3	140
8	0.5	4	120
9	1	1	120
10	1	2	140
11	1	3	80
12	1	4	100
13	1.5	1	140
14	1.5	2	120
15	1.5	3	100
16	1.5	4	80

Table 4:Constant parameters of injection molding process

Parameter	Injection temperature (℃)	Injection pressure (MPa)	Cooling time (s)	Mold temperature (℃)
Adjusted value	230	110	25	100

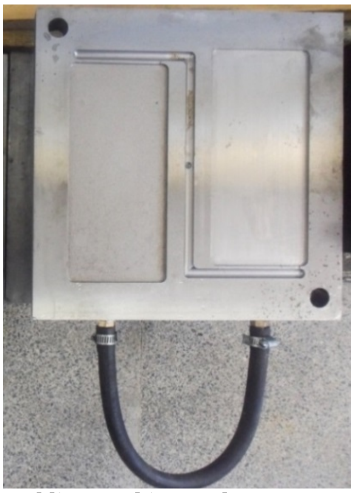
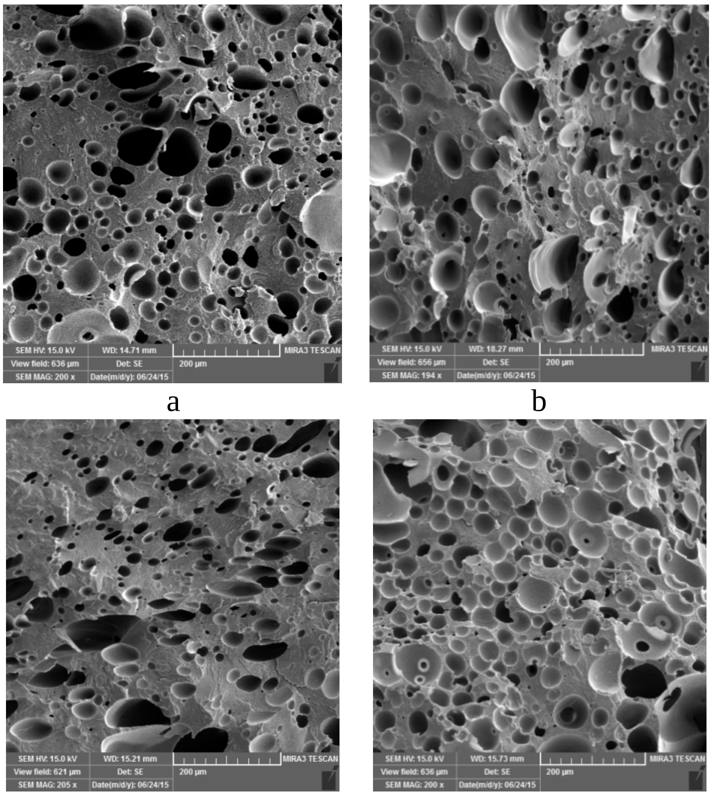


Fig.2.The mold of injection molding machine used to prepare nano composite foam plates

Fig.3.Samples of injected nano composite foam plates in various wt% of MWCNT

3. RESULTS AND DISCUSSION

The results of scanning electron microscopy (SEM) showed in Fig. 4. According these results, a good microcellular structure has been achieved.



a b c d

Fig.4. The results of SEM test for various conditions including a) foam of pure PA-6 in run 1, b) nanocomposite foam containing 0.5 wt% MWCNT in run 5, c) nanocomposite foam containing 1 wt% MWCNT in run 9 and d) nanocomposite foam containing 1.5 wt% MWCNT in run 13

3.1. Tensile strength

To investigate the mechanical properties of materials, perhapstensile testis the most important fundamental test of a material's mechanical response. One of the most important data's obtained from the tensile test is the ultimate tensile strength of the tested material. Tensile strength is the maximum tensile stress that a material can endure before it undergoes cracking, fracture or plastic deformation. [19]

In this research work specific tensile strength, symbolized STS, is defined as the ratio of tensile strength to the density of specimens tabulated in Table 5 For each run at least 3 samples were tested and the average of these three data reported as final specific tensile strength.

Importing data in Minitab software and analyzing them (according to larger is better state) will give the signal-to-noise ratio (S/N) plot of Fig. 5.Signal-to-noise ratio (S/N) values and importance rankings of the parameters according to amount of influence as represented in Table 6.

Table 5: Relative density of the examined samples

Sample	STS ($MPa. cm^3/g$)	Standard Deviation
1	14.42	0.819
2	13.93	0.644
3	15.72	0.856
4	16.78	1.051
5	19.74	0.714
6	21.57	0.908
7	27.64	1.106
8	31.03	1.261
9	38.09	0.740
10	34.18	1.142
11	36.98	0.614
12	40.94	1.291
13	36.35	0.891
14	29.72	1.146
15	33.79	1.321
16	39.29	0.836

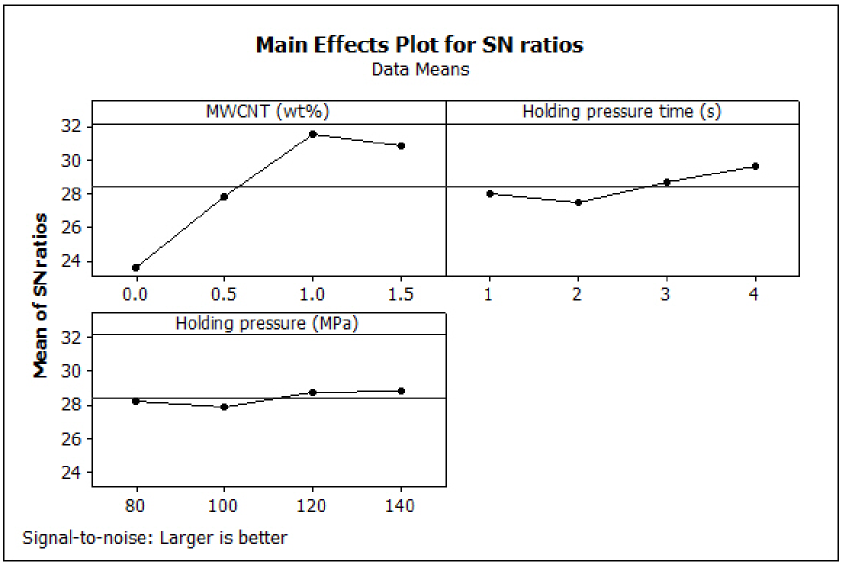


Fig.5.Signal to noise ratio plots for specific tensile strength of samples

Table 6:Signal to noise ratios for specifictensile strength and the importance ranking of parameters

Level	MWCNT (wt%)	Holding pressure time (s)	Holding pressure (MPa)
1	23.62	27.98	28.27
2	27.81	27.42	27.90
3	31.47	28.67	28.71
4	30.78	29.61	28.80
Delta	7.85	2.19	0.90
Importance Rank	1	2	3

According to Table 6and Fig. 5, weight percentage of MWCNT is the most effective parameter on specific tensile strength. The results indicated that by adding 0.5 and 1 wt% of MWCNTs, STS increases. The reason of this increase is the unique mechanical properties of MWCNTs. By addition 1.5 wt% of MWCNTs to polymeric matrix, the STS of samples decreased slightly in comparison to 1 wt% that the reason of this reduction is the agglomeration of MWCNTs in this level.The results also showed that injection processing parameters have low effect on STS.

3.3. Rockwell hardness test

Hardness test of specimens, as a suitable surficial mechanical property,are performed according to the Rockwell-R approach of hardness test [23, 24, 25]. For this purpose samplesare testedat least inthree points and the average quantity is reported as the final hardness number of the sample. In this research work specific Rockwell hardness, symbolized SRH, is defined as the ratio of Rockwell hardnessto the densityof specimens. Obtained results of specific Rockwell hardness is presented in Table 7.Analyzingthese datain Minitab software using, 'larger is better' state of Taguchi approach, leads to the signal-to-noise ratio values ofFig. 6 and Table 8. Importance ranking of parameters in terms of specific Rockwell hardness is also presented in Table 8.

According tothe results of thesignal to noise ratio weight percentage of MWCNT is the most effective parameter on the Rockwell hardness of samples. Results also indicate that as the amount of MWCNT increases, signal to noise ratio and consequently Rockwell hardness will increase. Obtained results of S/N ratio demonstrated that Level 4 (i.e. 1.5 wt% MWCNT) is the optimal level for weight percentage of nanoparticles. Results also reveal that holding pressure is the second effective parameter on Rockwell hardness of specimens. Similarly level 4 of holding pressure (i.e. 140 MPa) is the optimal level in terms of Rockwell hardness of specimens. Furthermore results revealed that holding pressure time has the lowest influence on Rockwell hardness, in a way that in higher levels of holding pressure time the signal to noise ratio and Rockwell hardness does not change significantly.

Table 7: Specific Rockwell hardness of specimens

Sample	SRH	Standard Deviation
1	89.65	0.54
2	94.66	0.69
3	97.94	0.14
4	103.88	0.54
5	102.78	0.33

6	**103.58**	**0.54**
7	**105.83**	**0.60**
8	**106.21**	**0.78**
9	**105.61**	**0.54**
10	**112.63**	**0.54**
11	**109.77**	**0.74**
12	**106.20**	**0.37**
13	**112.57**	**0.59**
14	**115.17**	**0.39**
15	**112.34**	**0.71**
16	**110.06**	**0.32**

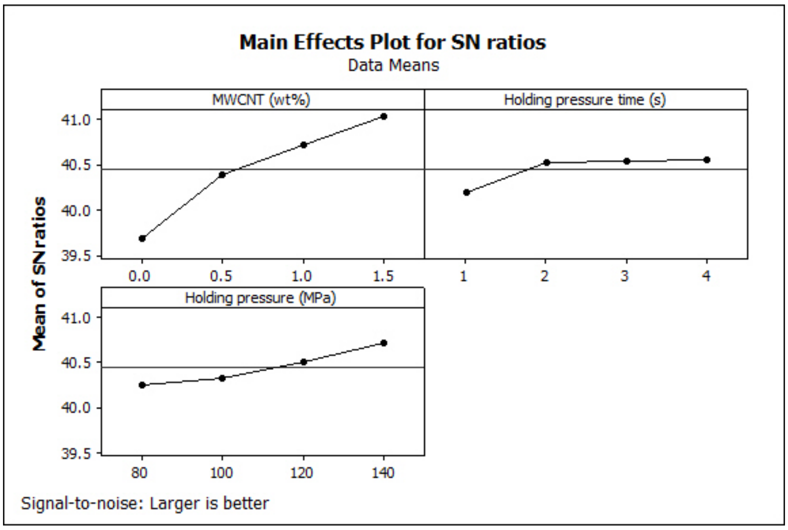


Fig.6. Signal to noise ratio plots for specific Rockwell hardness of samples

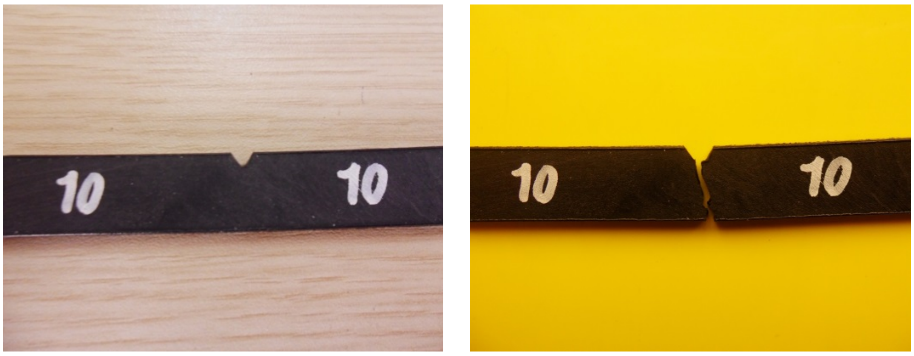
Table 8: Signal to noise ratios for specific Rockwell hardness and the importance ranking of parameters

Level	MWCNT (wt%)	Holding pressure time (s)	Holding pressure (MPa)
1	39.68	40.20	40.25
2	40.39	40.52	40.32
3	40.71	40.53	40.51
4	41.02	40.55	40.72
Delta	1.34	0.35	0.47
Rank	1	3	2

3.4

. Impact strength

Impact strength is one of the most important mechanical characteristics of a polymer that can introduce the toughness of materials. The impact strength is presented as the absorbed impact energy by the cross-section area of the samples. In present study, the samples are notched with 2 mm of depth, 45° of notch angle and 11.5*3.2 $mm^2$of cross-section area in order to perform Charpy impact tests. Fig. 7shows a sample of impact test before and after the test. In each run, at least 3 samples are tested and the average strength is reported as the final impact strength. The degree of Charpy impact test andspecific impact strength (the ratio of impact strength to density) of nanocomposite specimens are presented in Table 9. Based on the 'larger is better' state of Taguchi approach, signal to noise ratio analyses is presented in Fig. 8and Table 10. Importance ranking of effective parameters in terms of specific impact strength is also presented in Table 10.



a **b**

Fig.7. a) Prepared notched sample for Charpy impact test and b) tested sample

Table 9:Degree of impact test and specific impact strength of specimens

Sample	Degree	Standard Deviation	Specific impact strength (*$10^{-6} kj.m/g$
1	146.5	0.5	88.322
2	143.5	0.5	135.482
3	143	0.0	137.784
4	143.5	0.5	130.435
5	145	0.0	105.259
6	144.5	0.5	116.274
7	144	0.0	116.493
8	144	0.0	114.248
9	145.5	0.5	97.626
10	145.5	0.5	88.765
11	144.5	0.5	116.023
12	144	0.0	111.763
13	144	0.0	136.770
14	145.5	0.5	89.937
15	146	0.0	74.273
16	145.5	0.5	85.053

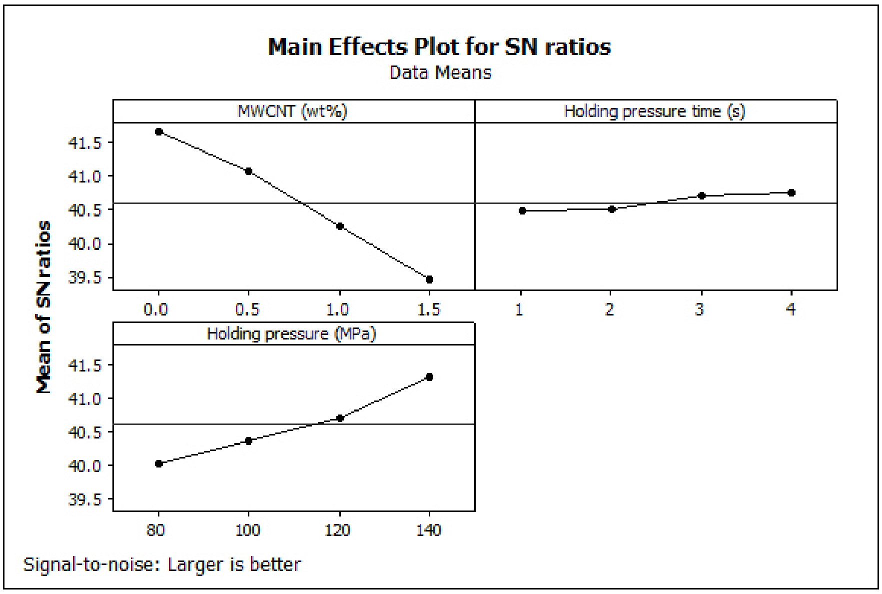


Fig.8: Signal to noise ratio plots for specific impact strength of samples

Table 10: Signal to noise ratios for specific impact strength and the importance ranking of parameters

Level	MWCNT (wt%)	Holding pressure time (s)	Holding pressure (MPa)
1	41.66	40.47	40.03
2	41.6	40.50	40.37
3	40.25	40.70	40.70
4	39.45	40.76	41.33
Delta	2.21	0.29	1.30
Rank	1	3	2

According to the signal to noise ratio analyses, the weight percentage of MWCNT is the most effective parameter on impact strength. As the results indicate when the weight percentage of MWCNT increases, signal to noise ratio and consequently impact strength decreases. In other word the impact strength of pure PA-6 foam samples is larger than those of nanocomposite foamed samples. Therefore the level 1 (i.e. pure PA-6) is the optimal level in terms of impact strength. Moreover the results of signal to noise ratio demonstrated that the second effective parameter on impact strength is holding pressure.Unlike to the weight percentage of MWCNT, if the holding pressure increases, signal to noise ratio and accordingly impact strength will be increased. Another important result that could be obtained from Fig. 12 is that holding pressure time does not have considerable effect on impact strength of samples.

4. CONCLUSION

In this research, first PA-6/MWCNT nanocomposites were foamed using Azodicarbonamidein an injection molding machine using a mold with dimension of

17.5*8 cm^2 and thickness of 3.2 mm. Then the nanocomposite foam samples were cut using a CNC laser cutting machine based on ASTM:D638 and ASTM:D6110 standard for tensile and impact test; respectively. Influence of the amount of MWCNT and injection molding parameters including holding pressure and holding pressure time on the mechanical properties of foamed samples have been investigated using L-16 orthogonal array of Taguchi method. After production the samples tensile strength, hardness and impact strength of samples were tested. According to the signal to noise ratio results, weight percentage of MWCNT is the most effective parameters on mechanical properties. Beside the results demonstrated that by adding 1 wt% of MWCNTs, specific tensile strength improved about 147%. Also adding 1.5 wt% of MWCNT increased the specific Rockwell hardness of samples about 17%. Obtained results also showed that by addition of MWCNTs, the specific impact strength of samples decreased.

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