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Utilization of Recycled Thermoplastic Nylon Combined with Virgin Nylon and the Effect on its Mechanical Properties as a Denture Base Material

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Abstract

The injection molding process of thermoplastic nylon produces nylon residues in the form of sprue. Nylon residues are non-biodegradable which causes serious ecological problems, hence recycling becomes a necessity. However, recycled nylon is subjected to thermal, oxidative, and mechanical degradation during process which may decrease the mechanical properties of recycled nylon. In order to overcome the decreased mechanical properties of recycled nylon, modification by combining recycled nylon with virgin nylon is considered.

Aim: This study aimed to determine the effect of adding virgin nylon into recycled nylon on the modulus of elasticity and compressive yield strength.

Settings and Design: Experimental laboratory research.

Methods and Materials: A total of 45 samples were used. Samples were divided into 3 groups which include 100% virgin nylon as control (A), 100% recycled nylon (B) and combination of 60% virgin nylon with 40% recycled nylon (C). The samples were tested using Universal Testing Machine (Tensilon RTF, Japan) with three point bending test and compression test with the speed of 5mm/min with ultimate load.

Statistical analysis used: The obtained results were analyzed using Univarian test, One-way ANOVA test and Turkey's Honestly Significant Different test.

Result: There was statistical significance in adding virgin nylon into recycled nylon on its mechanical properties, namely modulus of elasticity and compressive yield strength ($p<0.05$).

Conclusion: The combination of 60% virgin nylon with 40% recycled nylon has better elastic modulus and compressive yield strength values compared to 100% recycled nylon.

Keywords: Recycled nylon, Virgin nylon, Modulus of elasticity, Compressive yield strength.

Introduction

Thermoplastic nylon is a polymer belonging to the polyamide category and was introduced to the market for the fabrication of partial flexible dentures [1]. The demand for thermoplastic nylon dentures are very high because of its advantages such as its flexibility that increases the wearer's comfort, lightness, high aesthetic values because it does not have metal clasps and it is semi-transparent, very biocompatible because it has no residual monomers so it does not cause allergic reactions and it does not fracture easily [2-4]. On the other hand, thermoplastic nylon denture wearers do complaint about color changes due to the absorption of water, also thermoplastic nylon dentures tend to be not as retentive as it was initially (it becomes loose) due to the permanent deformation (compressive yield strength) of the non-metal clasps but the denture does not fracture clinically and is still intact [2,5].

Thermoplastic nylon has a low modulus of elasticity. It is very flexible and is indicated for patients with large undercuts. The more flexible a material, the longer it takes for the permanent deformation to occur due to the high compressive yield strength and vice versa, the more rigid the material, the lower the compressive yield strength. Compressive yield strength is the strength of a material to hold a load that exceeds the proportional limit or threshold value that causes permanent deformation but the structure or material is still intact and not fractured. Modulus of elasticity is the property of the material that allows it to change its shape if it is loaded and when the load is removed it will return to its original shape [6,7].

Injection molding technique is used to produce thermoplastic nylon denture base, but this manipulation technique causes the residual nylon in the form of sprue to be discharged after the polymerization process has been completed [8]. The accumulation of this non-biodegradable residual nylon has caused serious ecological problems due to the inability of the residual nylon to decompose over a period of time [9]. The effort taken to reduce environmental problems caused by the accumulation of residual nylon is to recycle the residual nylon mechanically. However, recycled nylon is subjected to thermal, oxidative, and mechanical degradation during processing which will decrease the mechanical properties of the recycled nylon, when the transition strain state of nylon is smaller, the crystalline degree decreases, hence the elastic modulus is increased and becomes more rigid resulting in brittle denture base. When crystalline degree of nylon increases, the elastic modulus will decrease so the denture base becomes more ductile or flexible, ie the compressive yield strength of the recycled nylon decreases and the modulus of elasticity increase [10]. To counter the decreased compressive yield strength and increased modulus of elasticity on the recycled nylon, it is modified by the addition of virgin nylon to the recycled nylon [9]. However, The percentage of virgin nylon should be higher than the percentage of recycled nylon in order to increase the molecular weight to get better mechanical properties [8,9].

Meyabadi et al recycled mechanically by modifying virgin nylon with recycled nylon which were mixed through a heating process with five different concentration ratios, with the ratios of 100%:0%, 75%:25%, 50%:50 %, 25%:75%, and 0%:100%. The results showed that the crystallinity of the ratio 50%: 50% is better than 75%:25% [8]. Maspoch et.al stated that there was a drastic decrease in the mechanical properties in recycled nylon which were not combined with virgin nylon. The researcher also stated that the percentage of recycled nylon must be lesser than virgin nylon and proposed that the ratio of 30% recycled nylon with 70% virgin nylon is the best ratio [11].

Based on the description that has been described, research has been conducted to determine the effect of adding 60% virgin nylon into 40% recycled nylon on the compressive yield strength and modulus elasticity of the thermoplastic nylon denture base, because higher percentage of recycled nylon than virgin nylon can affect the structure of combination nylon produced [12,13].

Materials and Method

This study is a laboratory experimental study with three different groups; 100% virgin nylon (group A), 100% recycled nylon (group B), and combination of 60% virgin nylon with 40% recycled nylon (group C). The samples used for elasticity modulus test were 45 rod shaped samples with dimension of 64 mm x 10 mm x 3.3 mm divided into 3 groups. Each group has 15 samples. The dimension of sample used for compressive yield strength was 10 mm x 10mm x 4mm ± 0.2 mm in accordance with ISO (International Standard Organization) no.604 [14]. The processing stage of nylon wastes are divided into four stages, which are the collection and sorting stage of nylon (sorting) in which the contaminants such as gypsum is sorted out, the cutting stage (shredding / cutting) in which the nylon is cut using a disc bur and also a cutter to equalize the size of nylon, washing stage to clean any residual contaminants using aquadest and drying stage to dry any excess water using a desicator (Duran, Germany) for 24 hours at 37°C. The nylon beads (BioPlast, USA) weighing 10 grams for 5 samples per injection using JET 7000 Injector W/ Moisture Filter (Snow Rock, USA) from each group was injected into a mold.

The samples were trimmed with tungsten carbide bur (Meisinger Lab TC, UK) and smoothed with waterproof sand paper (Kinik, Indonesia) with roughness of 600, 800, 1000 and 1200 mounted on a rotary grinder (Metaserv, England) to produce a flat surface and was continued with polishing with a Scotch-Brite brush (3M, USA) mounted on polishing motors (M2V Manfredi, Italy) and using coarse pumice (Kerr Dental, Swiss) until the samples were shiny. The compressive yield strength test was performed by compressive test and elasticity modulus test was performed with three point bending test and the two tests were conducted using Universal Testing Machine (Tensilon RTF, Japan). The elasticity modulus test is carried out by samples placed on the test apparatus, given a pressure bearing on it, then the load is given in the center continuously until the maximum load and cracking arises. The amount of deflection or flexure that occurs during the test is recorded on each load interval. The value of modulus of elasticity and its bending strength is calculated based on the maximum load, pedestal distance and cross-section. The compressive yield strength test is performed by providing the ultimate load-bearing cycle so that the sample fails or fractures and automatically stops the current to the motor. Data analysis was done by Univarian test, one way ANOVA test and Turkey's HSD test.

Results

Table 1 shows the elastic modulus values of three different groups, **Table 2** shows compressive yield strength values.

No.	Elastic Modulus (MPa)		
	Group A (Virgin nylon)	Group B (Recycled nylon)	Group C (Combination of 60% virgin nylon with 40% recycled nylon)
1	1965.98**	2633.70**	2300.56**
2	1965.98**	2629.41	2179.17
3	1894.49	2317.22	2063.21
4	1886.37	2263.38	2063.21
5	1781.54	2261.95	2007.42
6	1752.94	2196.45	1950.26
7	1724.35	1923.09	1837.30
8	1695.75*	1865.90	1752.94*
9	1726.32	1827.81*	1853.45
10	1761.38	2015.21	1983.42
11	1725.43	1987.63	1997.38
12	1850.23	2189.72	1949.25
13	1745.52	2042.78	1864.47
14	1783.58	2162.35	2010.23
15	1814.76	2089.62	1965.79

Note: * highest value, ** lowest value.

Table 1: Elastic modulus values of three different groups.

No. Sample	Compressive Yield Strength (MPa)		
	Group A (Virgin nylon)	Group B (Recycled nylon)	Group C (Combination of 60% virgin nylon with 40% recycled nylon)
1	80.162	46.576	75.871
2	78.909**	47.593	71.424**
3	82.216	47.749	77.291
4	82.149	47.328	74.943
5	82.896	49.727	76.436
6	81.532	45.417**	77.976*
7	79.533	46.432	76.274
8	83.575	50.298*	75.564
9	84.957*	49.904	75.730
10	82.365	49.652	75.905
11	82.753	48.615	74.552
12	83.645	47.896	74.468
13	81.972	47.351	73.570
14	82.561	46.278	75.821
15	82.237	46.892	75.625

Note: * highest value, ** lowest value

Table 2: Compressive yield strength values of three different groups.

The results of ANOVA test for elastic modulus and compressive yield strength were obtained =0.0001 ($p<0.05$). This means that there were statistically differences in elastic modulus values and compressive yield strength values between group A, group B and group C with significant value p =0.000 ($p<0.05$) **(Tables 3 and 4)**.

Group	Elastic Modulus (MPa)		
	n	$\bar{x} \pm SD$	p
A	15	1805.1+ 88.12	0.0001*
B	15	2160.4+241.29	
C	15	1985.2+136.28	

Note: * significant.

Table 3: The differences in the value of elastic modulus of virgin nylon, recycled nylon and combination of 60% virgin nylon with 40% recycled nylon.

Group	Compressive Yield Strength(MPa)		
	N	$\bar{x} \pm SD$	p
A	15	82.097 ± 1.580	0.0001*
B	15	47.891± 2.043	
C	15	75.723 ± 1.853	

Table 4: The differences in the value of compressive yield strength of virgin nylon, recycled nylon and combination of 60% virgin nylon with 40% recycled nylon.

Based on the result of Turkey's HSD test of the modulus of elasticity, there are significant differences in group A with group B with p = 0.0001 (p<0,05), group B with group C with p = 0,007 (p<0,05) and group A with group C with p value = 0,005 (p<0,05) showed significant differences. This means that there is an effect of adding 60% virgin nylon into recycled nylon **(Table 5)**.

Variable	n	Mean difference	p
Group A - B	15	355.44	0.0001*
Group A - C	15	180.22	0.005*
Group B - C	15	175.21	0.007*

Table 5: The effect of adding 60% virgin nylon into 40% recycled nylon on the elastic modulus of thermoplastic nylon denture base.

Compressive yield strength test analyzed by Turkey's HSD showed significant differences between group A with group B, group A with group C and group B with group C with p value = 0.0001 (p<0.05) **(Table 6)**.

Variable	N	Mean difference	p
Group A-B	15	34.183	0.0001
Group A-C	15	6.667	0.0001
Group C-B	15	27.516	0.0001

Table 6: The effect of adding60% virgin nylon into 40% recycled nylon on the compressive yield strength of thermoplastic nylon denture base.

Property Material Composition	Result		
	Group A	Group B	Group C
Highly volatile content moisture), wt%	0.6	0.5	0.5
Medium volatile content (polymeric material) wt%	98.8	97.4	97.7
Non-combustible matter (ash), wt%	0.2	1.3	1.0

Table 7: Thermogravimetry (TGA) Analysis.

Scanning Electron Microscopy (SEM)

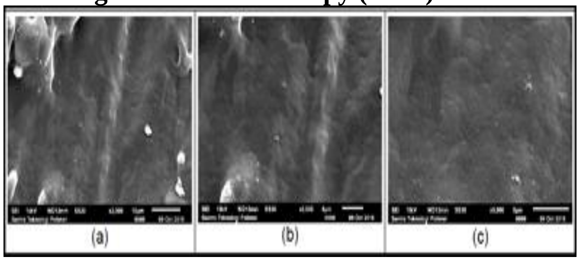

Figure 1: SEM Topography Sample of Virgin Nylon. (a) 2000x; (b) 3.000x; (c) 5.000.

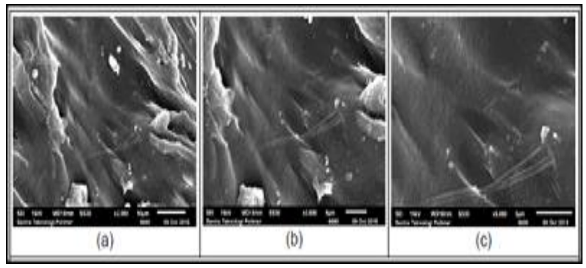

Figure 2: SEM Topography Sample of Recycled Nylon. (a) 2000x; (b) 3.000x; (c) 5.000.

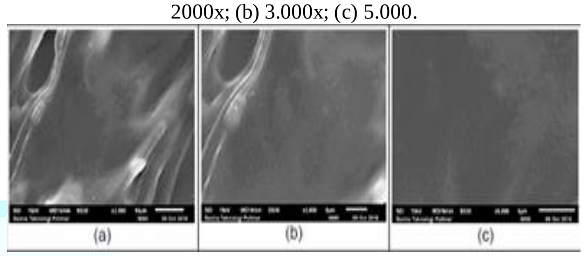

Figure 3: SEM Topography Sample of Combined Nylon. (a) 2000x; (b) 3.000x; (c) 5.000.

Discussion

Based on the results obtained from this research, the elasticity modulus value and compressive yield strength value varied on each sample in one group, although still in the scope of homogeneous data based on the homogeneity test. This might be due to the influencing factors during sample's fabrication process, among others, the possibility of porosity occurred during the injection molding process. Injection molding technique can produce porosity during the injection of thermoplastic nylon with injectors because air enters during the heating process. Porosity is divided into two categories, namely macro porosity and microporosity which requires Scanning Electron Microscopy (SEM) to detect its microporosity.

Mechanical properties of a material were affected by the porosity of the material because the higher the porosity of a material, the lower the mechanical properties of the material would be. The material used in this study was not fully dense because it was filled with air particles which resulted in porosity and would cause the material to easily deform and fracture as shown in figures 1,2 and 3 [15]. In this present study, macro porosity has been controlled by not using a sample that visually has porosity. In addition, to control the microporosity of this research samples, JET 7000 Injector W / Moisture Filter Injector (Snow Rock, USA) was used, which could reduce microporosity by creating a vacuum condition, hence air vapour might not be able to enter. The modulus of elasticity and compressive yield strength is also influenced by the degree of crystallinity. The higher the degree of crystallinity of a material, the higher the compressive yield strength, but the modulus of elasticity decreases [16].

Based on the results of one-way ANOVA test of elastic modulus and compressive yield strength, there was statistically differences of value in all three groups p = 0.0001 (p<0, 05). The differences between groups A, B and C might be caused by C-C chain scission reactions due to the mechanical recycling procedures through heating processes [5,6,8]. A virgin thermoplastic nylon group has a peptide unit (NH-CO) which produces a hydrogen bond between a stronger and more regular polymer chain resulting in a solid intermolecular space. However, in the recycled nylon group there is a C-C chain scission caused by the recycling process which will have an impact on the nylon's mechanical and physical properties [17].

The heating process that has been carried out on the recycled nylon group will produce water vapor, thereby increasing the nylon-bound H atom. The water molecule of H_2O has an H atom on its chemical element. Atom H will enter the chain and bind to the C atom on the nylon so that the initially long bond of CH_2-CH_2-CH_2 will be broken into CH_2 and CH_3-CH_3 so the number of CH3is more than CH_2. This causes the polymer chains formed on the recycled nylon group to become shorter and the chain bonds become weaker and irregular because the intermolecular spaces become less compact. As a result, the crystalline degree in the recycled nylon group is reduced and decreases the mechanical properties of nylon [18,19]. Similarly, Soja et al reported the effects of mechanical recycling on nylon waste. The results in FTIR showed that ratio of methyl (CH_3) increases, while methylene (CH_2) decreases as a result of chemical chain scission affecting nylon's mechanical and physical properties [9].

The Turkey's HSD test resulted from elastic modulus and compressive yield strength were similarly shown statistically significant differences in between group A with B, A with C and B with C. This could be stated that the addition of virgin thermoplastic nylon into the recycled nylon has an effect to the elastic modulus and compressive yield strength. This effect is due to the addition of a compatibilizer which is virgin nylon itself, into the recycled nylon causing the hydrogen bond in virgin nylon to fill the amorphous area of the recycled nylon causing the chemical chain to extend. In this present study, 60% of virgin nylon hydrogen bond could fill the amorphous region of 40% recycled nylon, hence the addition of virgin nylon into the recycled nylon had an influence on the compressive yield strength and elastic modulus and its value is almost as good as virgin nylon. Bucella stated that there was an effect of adding virgin nylon into recycled nylon due to the Densicalorimetry (DSC) used on the study shows a chain extension reaction which would result in a good chemical chain [20].

The modulus of elasticity and compressive yield strength is influenced by temperature, transition glass temperature (Tg), degree of crystallinity and molecular weight during the fabrication of nylon denture base, so it can change at any time, if one of the factor changes it's values, then the modulus of elasticity may also change. If the modulus of elasticity it may change and will affect the transition strain state of material when load is exerted. Hiroyuki stated that when the transition strain state of nylon was smaller, the crystalline degree was decreased, hence the elastic modulus was increased and become more rigid which resulted in brittle denture base [21].
When crystalline degree of nylon increased, the elastic modulus would decrease so the denture base became more ductile or flexible. When the elastic modulus was higher, the transition strain on the nylon became smaller thus resulted in the curve to increase rapidly when stress was applied but the strain did not change rapidly and therefore the nylon became brittle. When the elastic modulus was lower, the transition strain state increased with rapid stretching when stress was applied but the voltage had a slow rate of increase that caused the nylon to be flexible. The increasing flexibility of nylon will increase the value of compressive yield strength. The combination of virgin nylon with recycled nylon will increase the value of transition strain state which can increase the mechanical properties of the recycled nylon.

An ideal requirement of denture base material is rigid, but a rigid denture base will become brittle while the thermoplastic nylon is in great demand because it is good in aesthetic and flexible. The thermoplastic nylon has high flexibility because of its low modulus of elasticity which makes it resistant to fracture, also making it comfortable to wear and providing strong retention and can be used on areas with large undercuts. The high flexibility of this nylon makes nylon not suitable in free-ended edentulous cases without being combined with a metal frame, as it may cause leverage load to the adjacent tooth. The results of this present study revealed that the 40% recycled nylon combined with 60% virgin nylon had a higher modulus of elasticity value than virgin nylon but lower than recycled nylon. These results gave meaning in terms of ideal denture base terms.

Based on ISO, the ideal denture base requirement is to have a modulus of elasticity not less than 2000 MPA [8]. The result of this nylon combination is 1985.2 MPA close to the value of 2000 MPA. This means that combination of virgin and recycled nylon can be used for short free-ended edentulous cases, because the modulus of elasticity is lower but still has little flexibility. Similarly, the compressive yield strength of Group C is higher than Group B but lower than Group A, which means in terms of flexibility the combination nylon is more rigid than virgin nylon but more flexible than recycled nylon. The most common problem with thermoplastic nylon denture is the non-metal clasps, which often undergoes permanent deformation, and therefore the denture base is often loosened.

The results this research showed that Group C is slightly stiffer than Group A, and therefore this type of nylon can be used in cases of clasps that require strength but not in the undercut area. The denture base material that was produced from recycled nylon showed a high value of modulus of elasticity, which meant it, has the disadvantage of being brittle. It should be reminded that in combining virgin nylon with recycled nylon, the percentage of virgin nylon should be higher than the percentage of recycled nylon. Similarly, T Fattahi, concluded in his research that the percentage of combined virgin nylon should be higher than the recycled nylon in order to increase the molecular weight [8,9].

Conclusion

Within the limitations of this present study, it can be concluded that recycled thermoplastic nylon can be reused into a new denture base. However, one must pay attention to other properties such as physical, biological, chemical, and rheological properties as well as the requirements needed for the use as a denture base.

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