

The Effect of Thyme and Nigella Oil on Some Properties of Acrylic Resin Denture Base

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المخالصة

أهداف الدراسة تهدف الدراسة الحالية الى تقييم الصفات المفضلة، وتركيز زيوت حبة السوداء، و زيت الزعتر للإضافات ذات كلف مستوى واطئ والتوافق الحيوي وكواد مضاد حيوي مع مسحوق مادة راتنج أكريل طقم الأسنان الأساسي الحراري المتوفرة بشكل تجاري. **المواد والطرائق:** تم تحضير مائة وستون عينة من راتنج أكريل الحراري، الرئيسة هُباتُ بدون إضافات، وبالإضافات (زيت طبيعي بقي حبة السوداء وزيت الزعتر بالتركيز 1.5%، 1%، 0.5%، و 2%) لتقييم القوي المستعرضة، قسوة تللم، صفة اللون، monomer متبقي، دقة بعدية، مسامية، مقياس أطياف IR، وإختبارات حساسية ميكروبية مضاد جراثيمي. **النتائج:** أظهرت النتائج إختلاف معنوي ($p = 0.05$) وإضافة زيت حبة السوداء النقي السوداء وزيت الزعتر ادى إلى زيادة القوة المستعرضة، وقسوة مواد طقم الأسنان الأساسية، بدون فجوات، زيادة في تحرير monomer المتبقي في اليوم الأول، وكلتا الإضافات لزيت حبة السوداء النقي وزيت الزعتر يُفعّلان كالمواد المضادة للجراثيم. **الاستنتاجات:** مواد مضاف لزيت حبة السوداء النقي وزيت الزعتر بتركيز (1.5%) أوصيا لإغطاء الصفات المفضلة، وكعادة ميكروبي بعد مُعالجة قاعدة طقم أسنان راتنج الأكريليك، لكن زيت الزعتر لم يظهر أي تأثير على اللون قياساً إلى زيت حبة السوداء

ABSTRACT

Aims: To evaluating the preferable properties and concentrations of Genuine Nigella sativa and Thyme as oil low cost additive biocompatible antibiotic materials of denture base acrylic powder in relation to the commercially available heat cure acrylic resin material. **Materials and Methods:** One hundred and sixty acrylic resin Major heat cured samples were prepared without additives (Control), and with additives (A pure natural oil of Nigella sativa and Thyme 0.5%, 1%, 1.5%, and 2%) to evaluate transverse strength, indentation hardness, color property, residual monomer, dimensional accuracy, porosity, measurement of IR spectra, and anti-microbial-sensitivity tests. **Results:** Results showed a significant difference to $P= 0.05$. The addition of Thyme and Nigella oil increased the transverse strength, and hardness of the denture base materials, no porosities, an increase in residual monomer elution at 1st day, and both additives Nigella and Thyme oil act as antibacterial materials. **Conclusions:** The additives materials of pure natural oil of Nigella sativa and Thyme with concentration of 1.5% were recommended to give a proper properties, and as antimicrobial after curing of the acrylic resin denture base, but Thyme oil showed no effect on the color after curing in relation to Nigella sativa **Key words:** Nigella sativa, Thyme oil, acrylic resin denture base.

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INTRODUCTION

The heat cure denture base resins are extensively used for their good properties⁽¹⁾. Strength properties, accuracy, porosity, residual monomer and other properties are field for ongoing research, leading to various modifica-

tions to improve its strength and other properties either by different curing cycle^(2,3, 4, 5), deep freezing⁽⁶⁾, and or by using additives materials (glass flake polyethylene fiber, polybutene a reactive plasticizer)^(7,8,9, 10,11).

The aim of this study is to evaluate

the effect of preferable properties and concentrations of Genuine *Nigella sativa* and Thyme as oil low cost additive biocompatible antibiotic materials⁽¹²⁾ to denture base acrylic powder in relation to the commercially available heat cure acrylic resin material.

MATERIALS AND METHODS

Major base 2 heat curing resin (powder), and (liquid)[NIOM CE Lot ok 3799 ISO 1576 NIOM CE Lot ok 3799 ISO 1576] was used. A pure natural oil of *Nigella sativa* and Thyme was prepared to be used as additive antibiotic materials in concentrations (0.5%, 1%, 1.5%, and 2%).

A total of one hundred and sixty resin samples were prepared and divided into subgroups for each test as follows: Control Major Heat cured resin without additives, and eight groups of Major heat cured resin mixed with additive *Nigella sativa*, and Thyme oil in concentrations 0.5, 1, 1.5, 2% materials. Each group consists of eight samples. Heat cure polymer (P: L ratio of 3:1) was taken in, manipulated according to manufactures' instructions and the material was packed into the mould in dough stage. Care was taken to avoid porosities due to entrapment of air bubbles. Trail closure was performed. Curing of the samples were done by using a short curing cycle where the temperature was slowly raised to 73°C and held at 90 min. , followed by immersion in boiling water bath at 100°C for 30 min. according to manufacturer instructions. The samples stored in distilled water at 37°C for 48 h. before testing. All samples prepared in this study were evaluated for the presence of porosity by examining them under reflecting light microscope (LOMO Micmed 2), using X10 and X40 magnifications respectively^(19, 20) before being tested.

Evaluation of Transverse Strength: Samples were prepared in dimensions of 65×10×2.5±0.03mm (length, width

and thickness) respectively, for the transverse strength test (ADA specification No.12). The test was done in air by using a 3 points bending on an Instron universal testing machine (Wolpert, Germany). The device was supplied with a central loading plunger and two supports, with polished cylindrical surfaces of 3.2 mm in diameter and 50 mm distance between supports. The test samples held at each end of the two supports, and the loading plunger placed mid way between the supports. The samples were deflected until fracture occurred. The transverse strength was calculated using the following equation:–

$$\text{Transverse strength (N/mm}^2\text{)} = 3/2 \times P / bd^2 \text{ }^{(13)}$$

b– is the sample width (mm) *d*–is the sample thickness (mm); *I*– is the span length (mm; *P*– is the peak load (N)

Indentation Hardness Test: The samples were prepared with dimensions of 30×15×3±0.03 mm (length, width and thickness). The samples stored in distilled water at 37°C for 48hr. before testing. The polished surface was tested for hardness at five different locations then the mean is taken for each surface by using Rockwell hardness tester with an indenter in the form of round steel ball (6.350mm in diameter).

The sample was first subjected to a fixed minor load of 10kg, then load of 50kg was applied to the sample and the Rockwell hardness number was recorded after application of this load by 15 sec⁽¹⁴⁾.

Color Property Test: Samples were prepared with dimensions of 45×10×2.5 mm (length, width, and thickness) respectively according to spectrophotometric uses instructions.

The assessment of color property was performed using an ultra violet visible spectrophotometer (CECIL 2000). The absorbed light is measured with accuracy up to 0.001^(15, 16). The transmitted light can be calculated from the following equation:

$$A=2-\text{Log } T^{(17)}$$

A= Absorbance

T=Transmittance

Residual Monomer Test: Samples were prepared with dimensions of 20×20×3 mm (length, width and thickness) respectively. Each sample was introduced in a sealed glass flask containing 10 ml. of distilled water at 37°C. The samples were immersed for 24 h, 48h., 72h, 96h, 120h, 144h, and 168h, the supernatants were removed and replaced by 10 ml of fresh distilled water. The time dependence of the monomer concentration was followed by monitoring the amount of monomer present in the supernatant medium using a (CECIL 2000) ultraviolet-visible spectrophotometer ($\lambda=254$ nm)⁽¹⁸⁾.

A linear calibration curve of methyl methacrylate (MMA) concentration as a function of absorbance at 254 nm. was obtained using MMA standard aqueous solutions in the range 0.025–0.5 mg/ml. The results were expressed as a percent of released residual monomer mass with respect to the weight of the specimen.

Dimensional Accuracy: Samples were prepared in dimensions of 65×10×2.5±0.03mm (length, width and thickness) respectively according to (ADA specification No.12). Measurements on three dimensions were done by using digital caliper accuracy of 0.001mm.

Measurement of IR Spectra: Grinds about 5mg of the polymer to a very fine powder with a smallest possible drop of a suitable mulling agent Nujol(a mixture of paraffinic hydrocarbons). The spectrum of the mull is determined by placing it between two sodium chloride plates in the path of the sample beam⁽²¹⁾.

Anti-Microbial-sensitivity Test: Anti-Microbial-sensitivity test measure the ability and antimicrobial agents

to inhibit bacterial growth in vitro. This ability may be estimated by Disk-Diffusion of sensitivity to antibiotics⁽²²⁾. Prepare the media using Nutrient-Agar. Prepare the filter discs of heat cure methyl methacrylate before curing (6mm. Diameter) with additives 0.5% Thyme, and 0.5% Nigella. Transfer from primary culture that was taken from oral cavity mixture of bacteria (Gr⁻, Gr⁺) by using loop to test tube contain normal saline. After complete incubation, measure the zone of inhibition around the disc to determine the sensitivity.

Descriptive statistical analysis mean, standard deviation, one way analysis of variance (ANOVA), and Duncan's multiple analysis range tests were used in this study.

RESULTS AND DISCUSSIONS

Since the introduction of poly methyl methacrylate as a denture base material, it has suffered from having relatively poor mechanical properties.

The results of this study showed that the addition of Thyme and Nigella increased the transverse strength, and hardness of the denture base materials. This could be explained by water sorption phenomenon of methyl methacrylate denture base material^(23, 24), Nigella showed higher strength and surface hardness than Thyme, this could be due to chemical composition of the major unsaturated fatty acids were linoleic acid followed by oleic and lignoceric acids⁽²⁵⁾. The addition of Thyme oil and Nigella oil with concentration 1%, 1.5%, and 2% showed an increase in the hardness of the denture base, and no significance difference in between, this is due to unreacted monomer with coated polymer with oil(Tables 1 to Table 5).

Table (1): Mean and *t*- test of transverse strength of acrylic resin with additives.

Concentration	Thyme {Mean (N/mm ²)}	Nigella {Mean (N/mm ²)}	T value	P value
0.5%	62.25	106.5	3.335	0.045
1%	93.0	100.5		
1.5%	91.5	117.0		
2%	93.0	116.2		
0.5-2%	84.938	110.05	3.360	0.044
Control	81.999			

Table (2): One way ANOVA of the surface hardness test acrylic resin with additives.

Variable	SS	MS	F	P
Between acrylic resin & Thyme concentrations	194.50	27.79	8.66	0.000
Between acrylic resin & Nigella concentrations	104.25	34.75	18.95	0.001
Between acrylic resin Nigella & Thyme	57.58	19.19	4.19	0.047

SS: Sum of square, MS = Mean square, F = F value.

Table (3): Duncan's multiple analysis range test of the surface hardness of acrylic resin with Thyme material.

Sample (No.)	Variable (conc.)	Mean	SD.	Duncan's
C1	0.5% Thyme	110.00	1.73	B
C2	1% Thyme	114.33	1.15	C
C3	1.5% Thyme	118.00	0.00	D
C4	2% Thyme	116.00	1.73	CD
Control		108.99	1.35	A

SD: Standard deviation, conc.: concentration

Table (4): Duncan's multiple analysis range test of the surface hardness of acrylic resin with Nigella material.

Sample (No.)	Variable (conc.)	Mean	SD.	Duncan's
C5	0.5% Nigella	112.33	1.73	B
C6	1% Nigella	109.67	1.15	A
C7	1.5% Nigella	115.67	0.00	C
C8	2% Nigella	111.33	1.73	B
Control		108.99	1.35	A

SD: Standard deviation, conc.: concentration

Table (5): Duncan's multiple analysis range test of the surface hardness test of acrylic resin with additive materials.

Sample (No.)	Variable (concentration)	Mean	Duncan's
C1	0.5% Thyme	112.33	A
C2	1% Thyme	109.67	B
C3	1.5% Thyme	115.67	C
C4	2% Thyme	111.33	B
C5	0.5% Nigella	112.33	AB
C6	1% Nigella	109.67	A
C7	1.5% Nigella	115.67	BC
C8	2% Nigella	111.33	AB
Control		108.99	A

The results of amount of absorbance showed that mean differences for Nigella (0.406nm), while for Thyme was (0.378nm). This is due to natural findings of special change of the color of additive materials with Nigella (yellow), but colorless by using

thyme, these findings in agreement with Cheikh-Rouhou *et al.*,⁽²⁵⁾ they concluded that Nigella oil is more yellow-colored than other vegetable oils and they can protect against UV light (Table 6).

Table (6): Amount of absorbance (nm) of the samples.

Sample (No.)	Heat cure resin + additive material	Absorbance
(C1)	Heat cure resin+ 0.5% Nigella	1.778
(C2)	Heat cure resin+ 1% Nigella	1.760
(C3)	Heat cure resin+ 1.5% Nigella	1.720
(C4)	Heat cure resin+ 2% Nigella	1.728
(C5)	Heat cure resin+ 0.5% Thyme	1.750
(C6)	Heat cure resin+ 1% Thyme	1.830
(C7)	Heat cure resin+ 1.5% Thyme	1.360
(C8)	Heat cure resin+ 2% Thyme	1.628
Heat cure resin control		1.372
Mean differences of Nigella		0.406
Mean differences of Thyme		0.378

The addition of Thyme oil and Nigella oil with concentration 1%, 1.5%, and 2% showed higher residual monomer elution at 1st day, this could be due to unreacted monomer coated with oil, there is no previous studies in this field to correlate these results. This means that the elution of monomer was

higher at the 1st day then declines till the 7th day. The decrease in the daily release of the monomer occurred as a result of the diffusion of the monomer into water and by continuous polymerization promoted by the active radicals found in the polymer chain^(23,24) (Table 7).

Table (7): Mean percentage of amount of residual monomer (W/W %) of the samples.

No. sam- ple	1 st day %	2 nd day %	3 rd day %	4 th day %	5 th day %	6 th day %	7 th day %
C1	.19	.14	.03	.09	.03	.039	.02
C2	.16	.128	.02	.010	.03	.037	.029
C3	.162	.129	.03	.068	.03	.04	.027
C4	.161	.15	.034	.058	.03	.04	.031
C5	.158	.13	.029	.093	.027	.042	.032
C6	.156	.10	.02	.06	.02	.029	.025
C7	.101	.12	.04	.06	.039	.034	.03
C8	.152	.10	.027	.010	.026	.04	.03
Control	0.137	0.082	0.061	0.022	0.013	0.002	0.002

C1: Heat cure resin+ 0.5% Thyme, C2: Heat cure resin+ 1% Thyme, C3: Heat cure resin+ 1.5% Thyme, C4: Heat cure resin+ 2% Thyme, C5: Heat cure resin+ 0.5% Nigella, C6: Heat cure resin+ 1% Nigella, C7: Heat cure resin+ 1.5% Nigella, C8: Heat cure resin+ 2% Nigella

The results of mean differences of dimensional accuracy (thickness, length, and width) of acrylic resin denture base of control in relation to acrylic resin with additives Thyme and Nigella oil showed less effect (Figure 1

and Table 8). The range of dimensional change after the addition of oils was (0.234–0.368) within the range of conventional acrylic according to ANSI/ADA Specification NO. 12 (ISO 1567) for denture base resins.

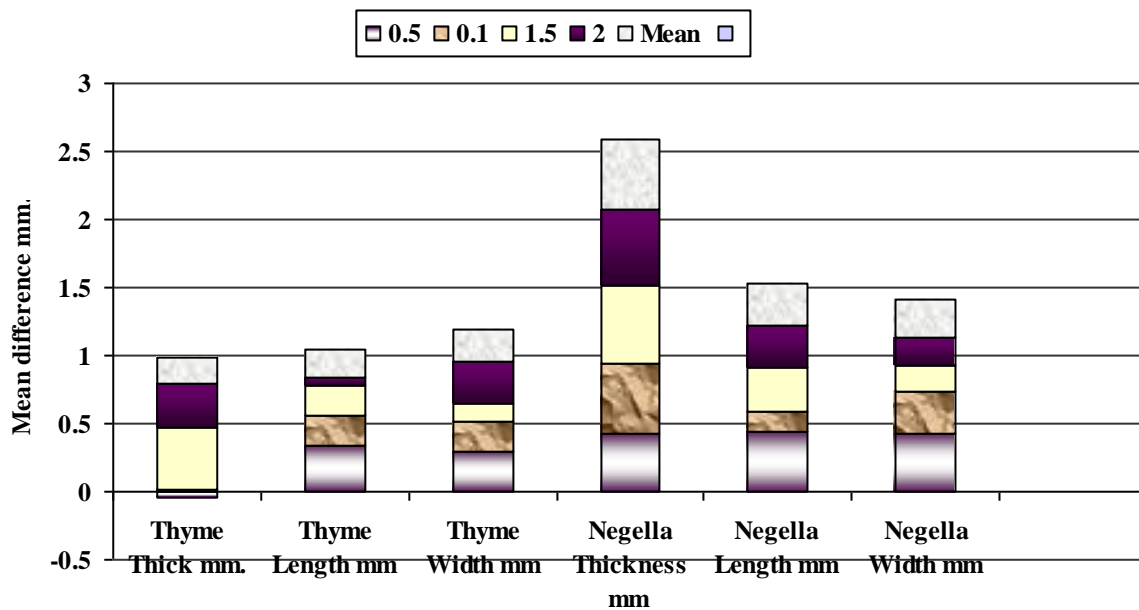


Figure (1): Mean differences in thickness, length, and width of acrylic denture base with additives in relation to conventional.

Table (8): Mean differences in dimensional change of acrylic resin denture base with additives in relation to the control.

Concentration	Thyme Oil(mm.)			Nigella Oil(mm.)		
	Thickness	Length	Width	Thickness	Length	Width
0.5%	-0.05	0.34	0.3	0.43	0.44	0.43
1%	0.02	0.22	0.22	0.51	0.15	0.3
1.5%	0.45	0.22	0.13	0.57	0.32	0.2
2%	0.33	0.06	0.3	0.56	0.31	0.2
Mean dimensional change	0.187	0.21	0.237	0.517	0.305	0.282

The results of antibacterial test showed that both additives Nigella and Thyme act as antibacterial material⁽²⁶⁾ (Figures 2 and 3). The effect zone of Nigella was bigger than Thyme. The effect of Thyme could be due to Phenol group, while the reason of antibacterial effect of Nigella oil might be due

to the complex chemical structure of the seeds. These little seeds have over than one hundred different chemical components, including abundant sources of all the essential fatty acids, though that is most often used medically. For example the essential oil of black cummin has an antimicrobial^(27, 28).



Figure(2): Antimicrobial test of Nigella oil.



Figure (3): Antimicrobial test of Thyme oil.

CONCLUSIONS

The additives materials pure natural oil of Nigella sativa and Thyme with concentration of 1.5% were recommended to give a acceptable properties, beside its antimicrobial effect after curing of the acrylic resin denture base, but Thyme oil showed no effect on the color after curing in relation to Nigella sativa.

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