



Effects of Neem Powder on Water Sorption and Solubility Characteristics of PMMA Denture Base Resin

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ABSTRACT

This study was conducted to determine the effect of neem (*Azadirachta indica*) leaf powder on the water sorption, water solubility and solvent resistance of heat-polymerised poly(methyl methacrylate) (PMMA) denture base resin. Neem leaves were sun-dried, pulverised and sieved to below 53 μm , and the powder was blended into PMMA polymer at 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0 g per 25 g of polymer (2 to 12 % of the polymer mass). Specimens were heat-polymerised alongside an unmodified control. Water absorption was determined as the percentage mass gain after 7 days of immersion in distilled water at 37 $^{\circ}\text{C}$, and solubility as the percentage mass lost after immersion and reconditioning to constant mass, on separate specimens. Resistance to water, acetone, ethanol, methanol, toluene and tetrahydrofuran was screened qualitatively. One specimen per formulation was tested for each property, and trends with loading were described by linear regression. All neem-modified specimens absorbed more water than the control (1.09 % to 1.68 %, compared with 0.21 %), although the control value was unusually low, so the size of the difference is uncertain. Absorption tended to increase with loading ($R^2 = 0.68$), but this trend depended largely on the specimen with the highest loading and was not confirmed by a rank-based test. Solubility ranged from 0.33 % to 1.43 %, compared with 0.58 % for the control, and showed no relationship with loading ($R^2 = 0.10$). All formulations, like the control, resisted water and the four organic solvents and dissolved only in tetrahydrofuran. Neem powder appears to increase the water uptake of PMMA denture base resin, and this should be confirmed with replicated, standardised specimens.

Keywords: denture base resin; PMMA; neem (*Azadirachta indica*); water sorption; solubility; natural additive

INTRODUCTION

Poly(methyl methacrylate) (PMMA) remains the principal material for denture bases because it is inexpensive, aesthetic and easily processed (Zafar, 2020). Its surface is readily colonised by microorganisms, and denture stomatitis, a chronic inflammation of the mucosa beneath the denture associated mainly with *Candida albicans*, is common among denture wearers (Gendreau & Loewy, 2011). Antimicrobial agents have therefore been incorporated into denture base resins (An, Evans, Hamlet & Love, 2021), but any addition to the resin can alter the properties on which the service life of the denture depends (Gad, Fouda, Al-Harbi, Năpănkangas & Raustia, 2017).

Neem (*A. indica*) is a tree of the family Meliaceae whose leaves contain limonoids, flavonoids and other constituents with broad-spectrum antibacterial and antifungal activity (Alzohairy, 2016; Wylie & Merrell,

2022). Neem powder has been incorporated into heat-polymerised PMMA as a natural antifungal additive (Hamid, Al-Dubayan, Al-Awami, Khan & Gad, 2019), and neem-modified PMMA has since been evaluated for antifungal activity after water storage (Saeed et al., 2024). Its effect on water sorption and solubility has received less attention.

Water sorption and solubility govern much of the long-term behaviour of a denture base. Absorbed water acts as a plasticiser in acrylic resin (Figuerôa et al., 2018), long-term water immersion lowers the flexural strength of heat-processed PMMA (Takahashi, Hamanaka & Shimizu, 2013), and water uptake is associated with dimensional change and discoloration, while the material lost to water consists of residual monomer and other leachable species with potential biological effects (Bettencourt et al., 2010; Goiato, Freitas, dos Santos, de Medeiros & Sonogo, 2015). A plant powder is a particularly relevant case, because dried leaf tissue is hygroscopic and enters the cured resin as unbonded particles whose interfaces with the matrix can provide paths for water (Gad et al., 2017). ISO 20795-1 accordingly sets upper limits for both properties in denture base polymers (International Organization for Standardization, 2013).

The aim of this study was to determine the effect of neem leaf powder, at loadings from 0.5 to 3.0 g per 25 g of polymer, on the water absorption, water solubility and solvent resistance of heat-polymerised PMMA denture base resin.

MATERIALS AND METHODS

Materials and powder preparation

Heat-polymerised PMMA denture base resin (Shanghai New Century Dental Materials Co., Ltd., China) was used for all specimens. Fresh neem leaves were purchased from Ekeonuwa Market, Owerri, Nigeria, cleaned to remove dust and foreign matter, and sun-dried until brittle and of constant weight. The dried leaves were pulverised in a mechanical grinder and passed through a standard test sieve of 53 μm aperture. Only the fraction passing the sieve was used. No solvent extraction was performed. The dried leaves and the sieved powder are shown in Fig. 1.



Fig. 1. Sun-dried neem leaves (left) and the sieved neem powder (right) used in this study.

Specimen fabrication

Seven formulations were prepared: an unmodified control and six neem-modified formulations (Table 1). For each modified formulation, the required mass of neem powder was added to 25 g of PMMA polymer powder and dry-mixed until the colour was uniform. Monomer was added at the manufacturer's polymer-to-monomer ratio of 2.5:1, and the mixture was allowed to reach the dough stage. The dough was packed into moulds prepared in Type III dental stone from stainless steel master dies, trial-closed under a hydraulic press and

finally closed. Flasks were held in boiling water for 20 min and allowed to cool in the bath. Specimens were deflasked, trimmed and finished with progressively finer silicon carbide papers. All masses were determined on an analytical balance with a readability of 0.001 g.

Table 1. Composition of the experimental formulations

Formulation	Neem powder (g)	PMMA polymer (g)	Neem (% of polymer mass)
Control	0	25	0
NE 0.5	0.5	25	2
NE 1.0	1	25	4
NE 1.5	1.5	25	6
NE 2.0	2	25	8
NE 2.5	2.5	25	10
NE 3.0	3	25	12

Monomer was added to each formulation at the manufacturer's polymer-to-monomer ratio of 2.5:1.

Water absorption

Specimens were conditioned in an oven at 37 °C and cooled in a desiccator until a constant initial dry mass (W_1) was obtained. Each specimen was then immersed individually in distilled water at 37 °C for 7 days. On removal, surface water was blotted off with absorbent paper and the specimen was weighed immediately to obtain the wet mass (W_2). Water absorption was calculated as $[(W_2 - W_1)/W_1] \times 100$.

Solubility

Solubility was determined on a separate set of specimens of each formulation. Each specimen was conditioned to a constant initial dry mass (W_1) as described in Section 2.3 and immersed in distilled water at 37 °C for 7 days. The specimens were then reconditioned in the oven at 37 °C, cooled in a desiccator before each weighing, and weighed at 24 h intervals until the mass was stable, which occurred at 72 h. The 72 h mass was recorded as the reconditioned mass (W_3), and solubility was calculated as $[(W_1 - W_3)/W_1] \times 100$.

Relation to ISO 20795-1

The immersion conditions followed ISO 20795-1 (International Organization for Standardization, 2013): specimens were immersed in distilled water at 37 °C for 7 days, and the solubility specimens were reconditioned to constant mass after immersion. The procedure departed from the standard in the respects listed in Table 2.

Table 2. Departures from the water sorption and solubility procedure of ISO 20795-1

Aspect	ISO 20795-1	This study
Specimens	Discs 50 ± 1 mm in diameter and 0.5 ± 0.1 mm thick	Dimensions not recorded; masses 0.72 to 2.41 g
Number tested	Replicate specimens of each material	One specimen per formulation for each property



Conditioning to dry mass	Desiccator at 37 °C until successive weighings differ by less than 0.2 mg	Oven at 37 °C, cooled in a desiccator; balance readability 1 mg
Sorption calculation	From the wet mass and the reconditioned mass of the same specimen	From the net mass gain (wet mass minus initial dry mass)
Specimens for sorption and solubility	The same specimens	Separate specimens
Units	$\mu\text{g}/\text{mm}^3$, from the measured specimen volume	Percentage of initial mass

Because of these departures, the results are reported as percentages by mass and are compared only with the unmodified control, which was prepared and tested in the same way. They cannot be used to judge compliance with the limits set by ISO 20795-1.

Solvent resistance

Small pieces of each cured formulation were placed separately in water, acetone, ethanol, methanol, toluene and tetrahydrofuran (THF) at room temperature and observed for dissolution. Each formulation was recorded as soluble or not soluble in each solvent.

Data analysis

One specimen of each formulation was tested for water absorption and one for solubility, so no formulation has a replicate and differences between individual formulations cannot be tested statistically. Results are reported as individual values and as the ratio to the control. The trend with neem loading was described by least-squares linear regression across the six neem formulations, using SciPy in Python 3 (Virtanen et al., 2020). Because each point is a single specimen, the regressions are descriptive and their p values are given for information only. The scatter of the six points about the regression line combines the variation between specimens with any departure from a straight-line trend, and its standard deviation was used as a rough estimate of the variation between specimens. The robustness of the trend was checked by repeating the regression without the highest loading and by Spearman's rank correlation with an exact p value. The readability of the balance (0.001 g) was used to calculate the largest error that rounding of the two weighings could introduce into each result.

RESULTS AND DISCUSSION

Water absorption

Every neem-modified formulation absorbed more water than the control (Table 3, Fig. 2). The control gained 0.207 % of its mass, and the neem formulations gained between 1.093 % and 1.683 %, from 5.3 to 8.1 times the control value. Absorption tended to increase with loading, from 1.093 % at 1.0 g to 1.683 % at 3.0 g, the highest value recorded, and linear regression across the six loadings gave a positive slope (0.177 percentage points per gram of neem, $R^2 = 0.68$, $p = 0.044$; Fig. 4a). This trend is not robust. It depended largely on the 3.0 g specimen: without it, the slope halved ($R^2 = 0.48$, $p = 0.20$), and Spearman's rank correlation across all six loadings was not significant ($\rho = 0.83$, exact $p = 0.058$). The 0.5 g formulation, at 1.243 %, lay above the regression line. The six neem values scattered about the regression line with a standard deviation of 0.13 percentage points, a rough estimate of the variation between individual specimens. The difference between the control and the lowest neem value, 0.89 percentage points, is about seven times this scatter, and it is larger than any error the balance could introduce: the largest rounding error in the two weighings would place the control between 0.17 % and 0.25 %, and the lowest neem value, at 1.0 g, between 1.03 % and 1.16 %.

Table 3. Water absorption of PMMA containing neem powder after 7 days in distilled water at 37 °C

Formulation	Initial mass W_1 (g)	Wet mass W_2 (g)	Water absorption (%)	Ratio to control
Control	2.409	2.414	0.207	1
NE 0.5	0.724	0.733	1.243	6
NE 1.0	1.554	1.571	1.093	5.3
NE 1.5	0.988	1	1.215	5.9
NE 2.0	1.119	1.134	1.34	6.5
NE 2.5	1.484	1.504	1.348	6.5
NE 3.0	1.248	1.269	1.683	8.1

One specimen per formulation.

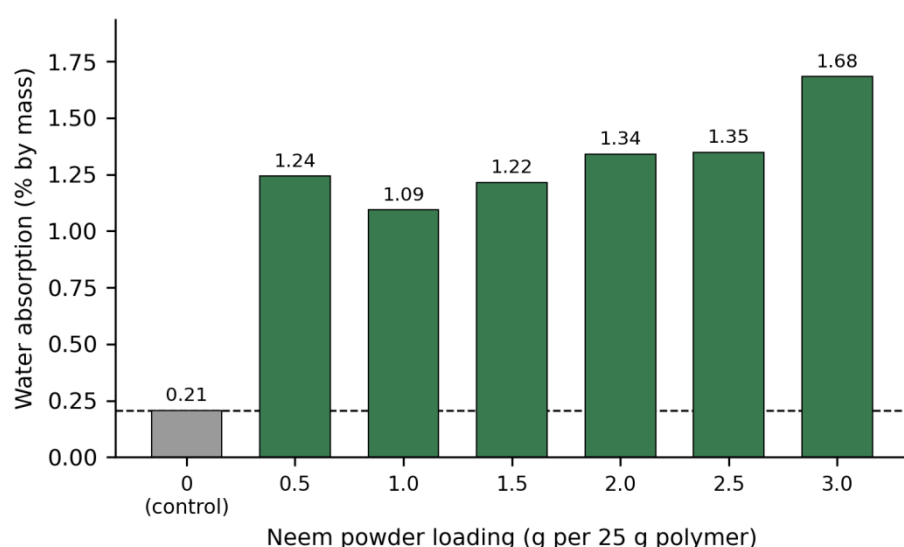


Fig. 2. Water absorption of PMMA containing neem powder after 7 days in distilled water at 37 °C. Dashed line, control.

The increase in water uptake with neem powder can be explained by two features of the additive. First, dried leaf tissue is hygroscopic, so each particle can itself take up water. Second, the powder was incorporated as whole plant material, without extraction, and entered the cured resin as unbonded particles. The interface between each particle and the matrix is likely to provide a path along which water can penetrate the resin; even in unmodified acrylic resin, water uptake depends on porosity and processing (Bettencourt et al., 2010; Figuerôa et al., 2018). Both the hygroscopic mass and the interfacial area increase with the number of particles present (Gad et al., 2017). The rise in absorption with loading is consistent with both mechanisms, although, as noted above, it rests largely on the 3.0 g specimen.

The value for the 0.5 g formulation, which was higher than those at 1.0 and 1.5 g, is best regarded as reflecting the variation between individual specimens rather than a real effect of the lowest loading. This specimen was the smallest tested (0.724 g), so that its 9 mg gain in mass represented a larger percentage than a similar gain in the heavier specimens, and a single-unit change in the balance reading altered its result by about 0.14 %. Every neem formulation nonetheless absorbed more water than the control. The control value of 0.207 % is, however, unusually low. Heat-polymerised acrylic resin immersed in water to saturation takes up about 2 % of its mass (Figuerôa et al., 2018). Seven days of immersion may not reach saturation, but the neem formulations, at 1.09 % to 1.68 %, lie closer to that figure than the control does. One likely reason lies in the method. Absorption was calculated from the net mass gain, so any material that leached out during immersion offset part of the

water taken up; ISO 20795-1 avoids this by calculating sorption from the reconditioned mass. If the mass lost by the solubility specimens (Table 4) is added back, water uptake becomes about 0.8 % for the control and 1.6 % to 2.7 % for the neem formulations, or 2.0 to 3.4 times the control. This estimate combines separate specimens and is indicative only, but it suggests that the ratios in Table 3 overstate the effect of neem, and the size of the increase remains uncertain.

The increase in water uptake is clinically relevant. Absorbed water acts as a plasticiser in acrylic resin (Figuerôa et al., 2018), and long-term immersion lowers the flexural strength of heat-processed PMMA (Takahashi et al., 2013), so neem-modified resins, which take up more water, may lose more of their mechanical performance in the mouth than their dry properties suggest. Increased water uptake is also associated with dimensional change and with a greater tendency to absorb stains.

Solubility

The control lost 0.58 % of its mass (Table 4, Fig. 3). Solubility of the neem formulations ranged from 0.33 % at 2.5 g to 1.43 % at 0.5 g. Four formulations (0.5, 1.5, 2.0 and 3.0 g) lost more mass than the control and two (1.0 and 2.5 g) lost less. Regression showed no significant relationship between solubility and loading ($R^2 = 0.10$, $p = 0.53$; Fig. 4b).

Table 4. Water solubility of PMMA containing neem powder after 7 days of immersion and reconditioning to constant mass

Formulation	Initial mass W_1 (g)	Reconditioned mass W_3 (g)	Mass lost (g)	Solubility (%)
Control	1.888	1.877	0.011	0.58
NE 0.5	1.815	1.789	0.026	1.43
NE 1.0	1.438	1.431	0.007	0.49
NE 1.5	1.284	1.273	0.011	0.86
NE 2.0	1.078	1.065	0.013	1.21
NE 2.5	1.204	1.2	0.004	0.33
NE 3.0	1.583	1.568	0.015	0.95

One specimen per formulation, separate from the specimens in Table 3.

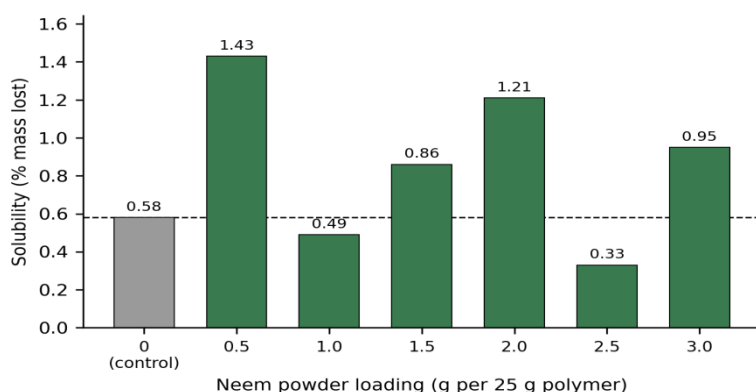


Fig. 3. Water solubility of PMMA containing neem powder after 7 days of immersion and reconditioning to constant mass. Dashed line, control.

Solubility did not follow the pattern of water absorption. The absence of a relationship with loading, and the fact that two neem formulations lost less mass than the control, indicate that the variation between individual

specimens exceeded any systematic effect of neem on solubility. The mass lost by the control, 0.58 %, is itself comparatively high. Since the control contains no additive, this points to factors common to all specimens, principally residual monomer leached from the resin (Rashid, Sheikh & Vohra, 2015). Figuerôa et al. (2018) found the highest solubility with their shortest polymerisation cycle and attributed it to a higher residual monomer content, and the short 20 min boiling cycle used here is likely to have had a similar effect. Some of the mass lost by the neem formulations is also likely to represent water-soluble constituents of the leaf powder. Leached species from denture base resins can affect the oral tissues (Goiato et al., 2015), so the nature of the material lost from neem-modified resin deserves further study.

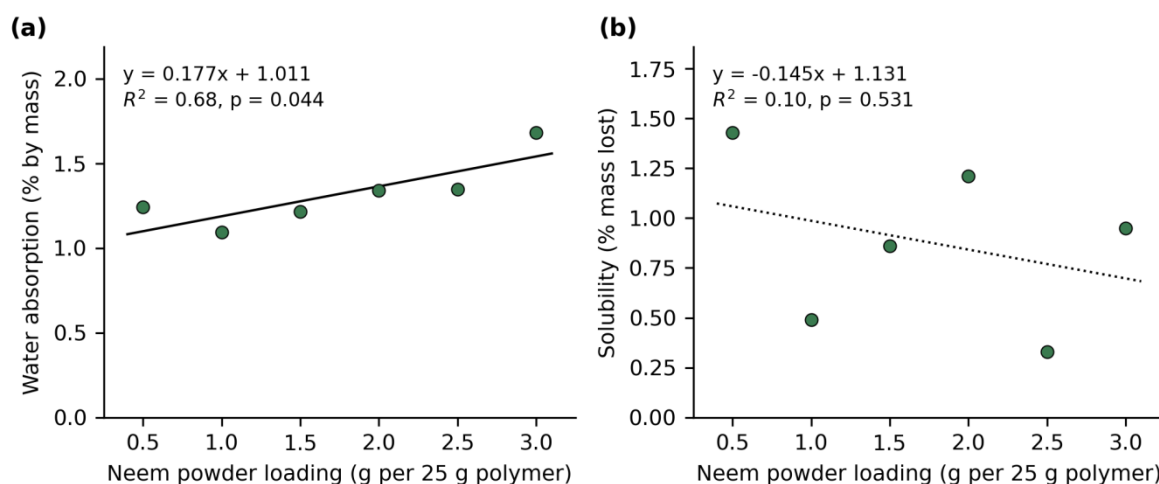


Fig. 4. Relationship between neem powder loading and (a) water absorption and (b) solubility, with least-squares regression lines. One specimen per formulation; the regressions describe the trend only and do not account for variation between specimens.

Solvent resistance

The control and all six neem formulations were insoluble in water, acetone, ethanol, methanol and toluene, and all dissolved in THF (Table 5).

Table 5. Solvent resistance of cured formulations at room temperature

Formulation	Water	Acetone	Ethanol	Methanol	Toluene	THF
Control	Not soluble	Not soluble	Not soluble	Not soluble	Not soluble	Soluble
NE 0.5 to NE 3.0	Not soluble	Not soluble	Not soluble	Not soluble	Not soluble	Soluble

THF, tetrahydrofuran.

The solvent screen shows that neem powder did not alter the chemical resistance of the resin. Every formulation behaved as unmodified PMMA, resisting water and four common organic solvents and dissolving only in THF, so the additive introduced no new vulnerability to solvents that a denture might encounter in cleaning or use.

CONCLUSIONS

This study examined the effect of neem powder on the water sorption and solubility characteristics of PMMA denture base resin. Within the scope of this in vitro study, neem leaf powder incorporated at 0.5 to 3.0 g per 25 g of polymer increased the water absorption of heat-polymerised PMMA denture base resin at every loading.



Because the control value was unusually low, the size of the increase is uncertain, and the rise in absorption with loading depended largely on the specimen with the highest loading. Neem powder had no consistent effect on water solubility and did not alter the resistance of the resin to common solvents. The increase in water uptake should be taken into account in selecting the loading of neem powder for denture base resins. Because these findings rest on one specimen per formulation and a procedure that departed from ISO 20795-1, they are preliminary and should be confirmed with replicated specimens prepared and tested to the standard.

DECLARATIONS

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Conflict of interest: The authors declare no conflict of interest.

Ethical approval: Not applicable; the study involved no human participants or animals.

Data availability: The data supporting the findings of this study are available from the corresponding author on reasonable request.

Author contributions: POO conceived the study idea. IOA designed the study. POO conducted the experiment, analyzed the data and wrote the draft manuscript. IOA and CO revised the manuscript and approved it.

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