

The effects of reinforcement with nanoparticles of polyetheretherketone, zirconium oxide and its mixture on flexural strength of PMMA resin

Purpose

Polymethylmethacrylate denture bases are prone to fracture, so reinforcement of dentures with nanoparticles is required to overcome these challenges. This *in-vitro* study was done to assess the effect of reinforcement with nanoparticles of polyetheretherketone (PEEK), zirconium oxide (ZrO₂) and its mixture on flexural strength of polymethylmethacrylate resin.

Materials and Methods

A total of 60 acrylic resin specimens measuring 65 mm × 10 mm × 2.5 mm were fabricated. The specimens were divided into fifteen specimens in each group [control group (C), 3wt% PEEK group (P), 3wt% zirconia group (Z), and hybrid reinforcement of 1.5wt% PEEK and 1.5wt% ZrO₂ group (P-Z)]. The flexural strength of the specimens was evaluated using a three-point bending test on a universal testing machine. The statistical analysis was done using one-way analysis of variance (ANOVA), and the intergroup comparison was done using Tukey's *post hoc* analysis.

Results

The mean flexural strength was maximum in group P-Z (98.73MPa) followed by group P (86.22 MPa) and group Z (84.48 MPa). The mean flexural strength was least in the control group (74.86MPa). One-way ANOVA revealed a highly significant (P<0.01) difference among the groups. Pairwise comparison among groups showed a significant difference (P<0.05) among all the groups except in between groups P and Z where no significant difference was found (P=0.406).

Conclusion


Hybrid reinforced PEEK and zirconia could be used as an effective reinforcement material for denture base resin. The hybrid PEEK and zirconia reinforced resin can be an alternative treatment option in patients with heavy occlusal forces and for patients who have previous experience of multiple denture fractures.

Keywords: Flexural strength, nanoparticles, polymethylmethacrylate, polyetheretherketone, zirconia

Introduction

Polymethylmethacrylate (PMMA) contributes up to 95% for the fabrication of removable dental prosthesis, due to its optical properties, biocompatibility, and aesthetics (1,2). However, significant issues still exist, which need to be addressed to improve the properties of PMMA for fabrication of dentures. PMMA denture bases are more prone to fracture due to stress concentration at the frenum notch, in rugae areas, in denture base regions with scratches, and under heavy masticatory forces (3-5).

Two methods were recommended to prevent the denture base fracture, one is by reinforcement of the denture base material and the oth-

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er is by reducing the stress concentration in the midline of the denture base (6). Various denture base designs/techniques have advocated to decrease the stresses at the midline, but this usually increases the denture base thickness with decrease in the tongue space and thus influences the speech (7). Incorporation of nanoparticles to increase the strength of the denture bases surely emerge as a better treatment option.

Numerous endeavours seemed to be attempted in the past to enhance the mechanical properties of the acrylic resins by incorporating different strengthening materials: metal fillers, metals, carbon fibres, aramid fibres, glass fibres and ultra-high molecular weight polyethylene (8). Although the incorporation of fibres improves the flexural strength, increased fibre content generally decreases the surface hardness without much increase in strength (9-11). The expansion of metal fillers increases the compressive strength and thermal conductivity but compromises the esthetics and decreases the tensile strength (12).

For the past few years, zirconia has been used to strengthen the denture bases. Zirconia is a white crystalline dioxide of zirconium, with a flexural strength of 1666 MPa and having a modulus of elasticity like steel (13). Zirconia incorporated in dental materials has enhanced the mechanical properties of the dental materials with better esthetics (14-16). In recent years, polyetheretherketone (PEEK), a semi-crystalline linear polycyclic aromatic polymer, is used frequently in dentistry (17,18). PEEK is non hypersensitive and has low plaque affinity, with a flexural modulus of 140-170 MPa (18,19). Young's modulus and tensile properties of PEEK are similar to human bone, enamel and dentin (20,21). PEEK material is one of the better esthetic material utilized for the manufacture of removable partial dentures.

The present study was aimed to enhance the flexural strength of denture base resin by reinforcing it with PEEK and zirconia. Currently, no literature is available utilizing the hybrid reinforcement of PEEK and zirconia in acrylic denture base resin. This in vitro study was done to evaluate the flexural strength of PMMA denture base resin reinforced with 3wt% PEEK, 3wt% Zirconium oxide (ZrO_2) and in combination with 1.5wt% PEEK and 1.5wt% ZrO_2 . The null hypothesis in the study was that there would be no difference in flexural strength of reinforced denture base resin with 3wt% PEEK, 3wt% ZrO_2 and with a mixture of both 1.5wt% PEEK and 1.5wt% ZrO_2 when compared to non-reinforced denture base resin.

Materials and methods

This in vitro study was done in the Prosthodontics Department with technological aid from the Central Institute of Plastics Engineering and Technology (Bhopal, India) and the Centre for Scientific Research and Development (Bhopal, India). A total of sixty specimens were made, with each group containing 15 specimens. The specimens were broadly divided into two groups: n=15 control group (C) and n=45 experimental groups (E). The experimental group is further divided into 3 subgroups with n=15 specimens each (Figure 1).

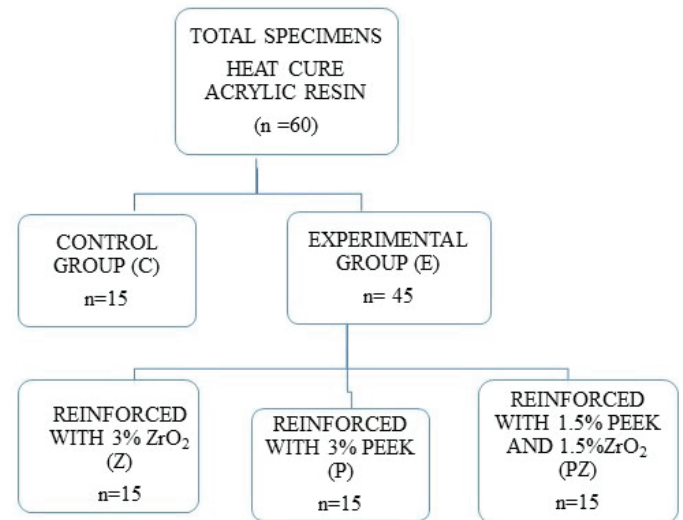


Figure 1. Flow chart depicting distribution of specimens.

Specimen fabrication

Wax specimens with dimensions of (65mm length x 10mm width x 2.5 mm thickness) were fabricated in a hard plastic mold according to American Dental Association specifications No.12 (Figure 2) (22).

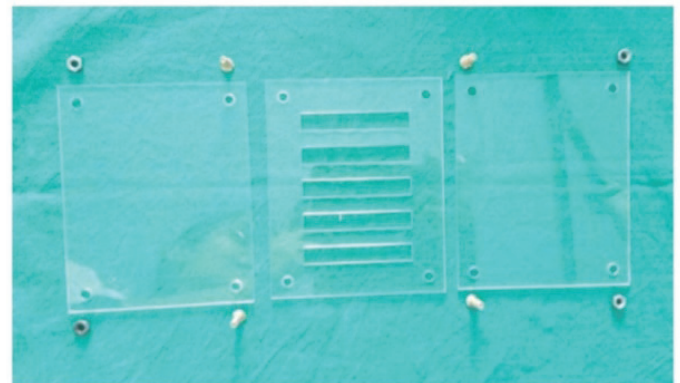


Figure 2. The three piece standardized mold.

The middle part of the mold was assembled over the lower part and petroleum jelly (Unilever, Mumbai, India) was applied. The mold was filled with the softened modelling wax (DPI, Mumbai, India). The cover plate was positioned in place and tightened with the screws to eliminate the extra wax. After some times, as the wax solidifies, the cover plate was unscrewed and the surplus wax was eliminated with a Bard Parker blade (Sigma Aldrich, New Delhi, India). The wax specimens were retrieved from the mold. The wax specimens, which were uniform in all dimensions, were taken for flasking and distorted specimens were eliminated. The specimens were invested in dental stone (Kalrock, Kalabhai, Mumbai, India) in flasks and allowed to set for 1h (Figure 3).

The flasks were kept in the dewaxing unit for 8 min and then opened and any residual wax was flushed by spraying with hot water. The mold was coated with separating medium (Coe-Sep, GCAcro-Sep, Europe). The required amount of PMMA, ZrO_2 and PEEK required to be mixed with acrylic resin was measured with an electronic balance having precision of up to three decimal places.



Figure 3. Wax specimen positioned in the mold.

Control group specimens

Control group specimens were fabricated with heat cure PMMA resin (Trevalon HI, Dentsply, Mumbai, India) incorporated in the ratio of 21 g polymer:10 ml monomer.

Experimental group specimens

PEEK group (P) specimens were fabricated with 3wt% PEEK (Vivtrex PEEK, Padmini Innovative Marketing Solution Pvt. Ltd. Mumbai, India) in ratio of 0.630g PEEK:20.370g polymer:10 ml monomer. Zirconia group (Z) specimens were fabricated with 3wt% ZrO_2 powder (Yttria stabilized zirconia nanopowder, Nanosheel Creating Miracles in black, Willmington DE, USA) in a ratio of 0.630g ZrO_2 :20.370g polymer:10 ml monomer. For specimen fabrication of a combination group of PEEK and ZrO_2 (P-Z), 1.5wt% PEEK and 1.5 % ZrO_2 powder in a ratio of 0.315g PEEK:0.315g ZrO_2 :20.370g polymer:10 ml monomer was used (Figure 4). A uniform mixture of the PEEK/ ZrO_2 /combinations within the acrylic powder was obtained with a blender running at a speed of 400 rpm for 30 min.



Figure 4. Zirconium oxide and PEEK powder.

Processing of specimens

Specimens were packed in the mold in the dough stage and the flask closed together. The packed flasks were kept under a hydraulic press (Mestra 48150 Sondika-Bilbao, Spain) applying a pressure of 14MPa for 30min. Conventional heatcure polymerization procedure was carried out for these packed specimens under a water bath for 9 h [(7 h/74 °C ($\pm 3^\circ\text{C}$)) followed by 2 h/95°C ($\pm 3^\circ\text{C}$)]. After completion of the curing cycle, flasks were kept for 30min at room temperature for cooling, followed by cooling for 15mins under running tap water. The flasks were opened and specimens were retrieved. Finishing of specimens was done followed by polishing with silicon carbide paper of different grids (1000, 800, and 600 coarseness) (Figure 5). All the specimens were stored in an incubator containing distilled water for 48h at $37^\circ\text{C} \pm 1^\circ\text{C}$. To check the uniform dimensional accuracy in all the specimens, digital vernier calliper was used to measure at three different areas with a tolerance of not more than 0.2mm dimensional discrepancy.

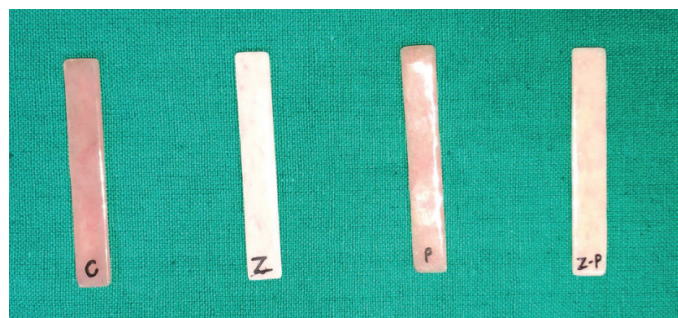


Figure 5. Fabricated specimens.

Three-point bending test

The specimens were placed under universal testing machine (Instron Corporation, Canton, MA, USA) (Figure 6) for 3-point bending test and flexural strength was evaluated at a crosshead speed of 2 mm/min. The fracture load (peak load)

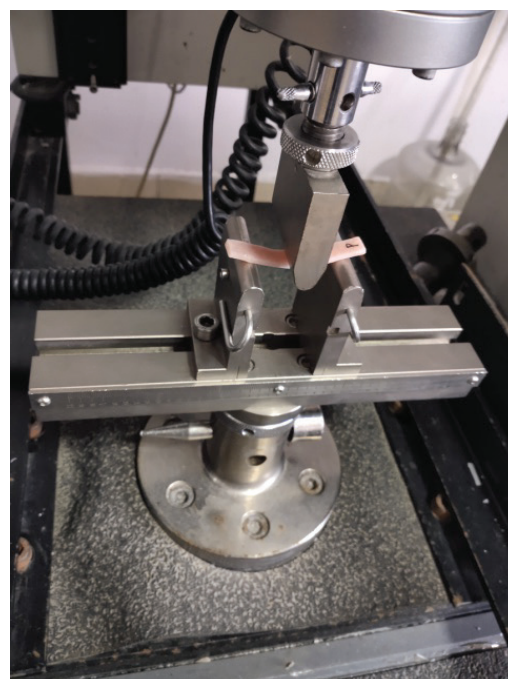


Figure 6. Specimen under 3-point bending test.

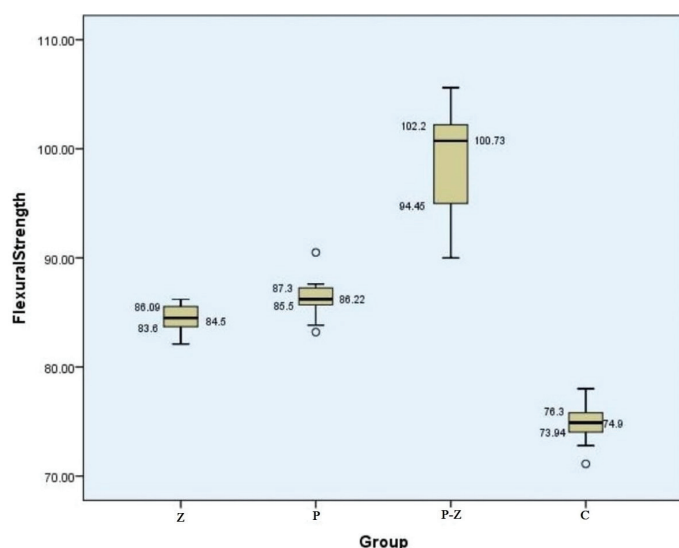


Figure 7. Box plot showing flexural strength at maximum load of samples in different groups.

for each specimen was evaluated and converted to flexural strength by using the formula $S = 3PL/2bd$ Where S =flexural strength (N/mm²); P =load at fracture; L = distance between jig supports; b =specimen width; d =specimen thickness.

Statistical analysis

The data obtained was subjected to statistical analysis using Statistical Package for the Social Sciences (IBM Corp. Released 2011. IBM SPSS Statistics for Windows, Version 20.0. Armonk,NY:IBM Corp.).The statistical analysis was done using one-way analysis of variance (ANOVA), and the inter-group comparison was done using Tukey's *post hoc* analysis. A P-value<0.05 was considered statistically significant. The confidence interval was set at 95%.

Results

The mean flexural strength of the control group and experimental groups were presented in Table 1. The mean flexural strength of the experiment groups was significantly higher ($P<0.05$) than the control group. The mean flexural strength was maximum with a mixture of 1.5wt% PEEK and 1.5wt% ZrO₂ (98.73MPa) followed by 3% PEEK (86.22 MPa) and 3%ZrO₂ (84.48 MPa). The mean flexural strength was least in the control group (74.86MPa) (Fig 7). One-way ANOVA revealed a highly significant ($P<0.01$) difference among the groups. Pairwise comparison among groups showed a significant difference ($P<0.05$) among all the groups except

in between the 3% PEEK group and 3% zirconia group, where no significant difference was found ($P=0.406$) (Table 1).

Discussion

The null hypothesis was rejected. Polymethylmethacrylate dentures are vulnerable to fracture while use or when accidentally dropped onto any hard surface due to their flexural fatigue on a time (9). Flexural fatigue generally happens due to continuous flexing of dentures which leads to the development of microcracks in the stress concentration area. Midline cracks are a typical issue for patients with maxillary complete dentures, due to cyclic disfigurement resulting in flexural fatigue (23). Despite the recent trend of incorporating ceramic fillers and composite materials into denture base resins, it is required to understand the effects of hybrid reinforcement of PEEK and zirconia in denture base resins. Muhsin *et al.*(24) determined the mechanical properties of PEEK polymer when used as a denture material in their study and found PEEK material to be a resistant material to notch concentration. They stated that if PEEK material is used for denture frameworks having notches at labial or buccal frenum, in these conditions too they are less prone to fracture. When used in elastic region PEEK has increased tensile strength with less plastic deformation compared to PMMA. In the present study, an increase of flexural strength (86.2MPa) by 15.17% was present with PMMA filled with PEEK filler which was more compared to the control group (74.8 MPa). PEEK can be advantageous in reducing stress concentration at notches for labial or buccal frenum, if incorporated as nanoparticles in denture bases.

Zidan *et al.*(25) had analysed the flexural strength of high impact heat-polymerised PMMA resin incorporating various concentrations of ZrO₂ nanoparticles (1.5%, 3%, 5%, 7%, and 10wt%).They found that the inclusion of ZrO₂ nanoparticles in PMMA resin had gradually increases the flexural strength up to 3 wt% and after that the flexural strength decreases at higher concentrations when compared to the control group. There was a 15% significant increase in flexural strength and it was highest in the group having 3 wt% ZrO₂ (83.5 MPa) when compared to the control group (72.4 MPa). Filler concentration at 3wt% seems to increase the flexural strength. Specimens with high filler concentration causes more filler-to-filler interactions compared to matrix-to-filler interactions and forms an agglomeration causing non uniform stress distribution due to forming a point of stress concentration (26). In the present study, a similar result was found with an increase of 12.85% in flexural strength when PMMA was strengthened with 3wt % ZrO₂ (84.4 MPa) in comparison to

Table 1: One way ANOVA for flexural strength of different groups.

Group	n	Mean (MPa)	SD	F ratio	P value	Tukey's post hoc analysis
Control group (C)	15	74.86	1.74			$P_1<0.05^*$
3wt% Zirconia group (Z)	15	84.48	1.34			$P_2<0.05^*$
3wt% PEEK group (P)	15	86.22	1.78	46.868	<0.0001	$P_3<0.05^*$
Hybrid reinforcement of 1.5wt% zirconia 1.5wt% PEEK group (P-Z)	15	98.73	4.97			$P_4=406$
						$P_5<0.05^*$
						$P_6<0.05^*$

*P value<0.05 was considered statistically significant. ANOVA=Analysis of variance; P1= between group C and group Z; P2= between group C and group P; P3= between group C and group P-Z; P4= between group Z and group P; P5= between group Z and group P-Z; P6= between group P and group P-Z

the control group (74.86 MPa) and the difference obtained was statistically significant. Zirconium oxide as nanoparticles has a large interfacial area which enhances the contact points in between the PMMA and ZrO_2 , thus promotes additional mechanical interlocking and with more flexibility (27).

Sirandoni *et al.* (28) in a 3D finite element analysis, evaluated the biomechanical properties of various framework materials used for fabrication of implant supported mandibular fixed prosthesis. They favoured zirconia material over PEEK and PMMA as a framework material. Muhsin *et al.* (24) found in their study that PEEK had a higher tensile strength than PMMA and could be preferred for fabrication of denture in the near future. Thus, we reinforced both ZrO_2 and PEEK into PMMA to incorporate the qualities of both the materials. Gad *et al.* (29) in their study reinforced the PMMA resin with ZrO_2 nanoparticles and glass fibers (GFs) in different concentrations and found increased flexural strength of group with 2.5% ZrO_2 + 2.5% GFs by 45% compared to that of non-reinforced PMMA. The increase in flexural strength was possible because of the synergistic effect of ZrO_2 and GFs. In present the study, the hybrid reinforcement of PEEK and ZrO_2 (1.5wt% PEEK and 1.5wt% ZrO_2) in PMMA was done and a 31.88% increase in flexural strength (98.73MPa) was found compared to non-reinforced PMMA. In the present study maximum flexural strength found in the hybrid group which may be due to the synergistic effect of PEEK and ZrO_2 .

In this study, to enhance the mechanical bonding of the PEEK and zirconia with PMMA, the powders were mixed with a blender running at a speed of 400 rpm for 30 min. This process helps in achieving an even distribution of the PEEK/ ZrO_2 combinations within the acrylic powder. This helps in better bonding and reducing the agglomeration tendency in the mix and thus helps in reducing the points of stress concentration (29).

No difficulty was encountered during the finishing and polishing of the specimens and a well-polished surface was obtained with the specimens of all the groups. The shade obtained with the ZrO_2 group has a more whitish appearance when compared to other groups. The shade obtained with the PEEK group was almost similar to the control group. The shade obtained with the PEEK- ZrO_2 group was slightly whiter compared to the control group but seems esthetically acceptable.

One of the basic requirements for a successful denture is flexural strength, which should be enough to prevent catastrophic failure under loading (20,30,31). A completely polymerized acrylic resin has better mechanical properties (32,33). The increased flexural strength indicates the quality of polymerization and suggests that the denture can resist the applied forces. In the present study, the overall result showed that the flexural strength of hybrid reinforced PEEK and zirconium oxide with denture base material has higher flexural strength than PEEK and zirconia individually with denture base material. The hybrid reinforcement might be helpful in bruxism patients, and in patients with resorbed ridge who are more prone to denture fractures. In patients with prominent anterior maxilla, the hybrid dentures can be given with thinner flanges as an alternative to flangeless denture with acceptable esthetics and at an affordable cost.

The limitation of the present study is that the study is *in-vitro* which is commonly performed to predict the behavior of materials in the clinical setting, but it would have provided further information if thermo-cycling would have been

done to better simulate the oral conditions. Further research simulating the oral conditions is required to investigate the performance of this material in present and other possible combinations to find out whether they had any effect on other mechanical and physical properties of the denture bases. Scanning electron microscope study should be done to find the surface characteristics, distribution of nanoparticles in the mixture and to check for porosities and formation of agglomerates at the fracture site.

Conclusion

Hybrid reinforced PEEK and zirconia could be used as an effective reinforcement material for denture base resin. Hybrid PEEK and zirconia reinforced resin can be an alternative treatment option in patients with heavy occlusal forces and for patients who have previous experience of multiple denture fractures. Further studies are required to test the performance of this combination in fatigue testing and cyclic loading to establish the result of the present study.

Türkçe Özet: Polietereterketon, zirkonyum oksit ve karışımının nanoparçacıkları ile güçlendirmenin polimetilmetakrilat reçinesinin eğilme mukavemeti üzerindeki etkisi. Amaç: Polimetilmetakrilat protez kaideleri kırılmaya eğilimlidir, bu nedenle protezlerin nanoparçacıklarla güçlendirilmesi gerekebilir. Bu *in vitro* çalışma, polietereterketon (PEEK), zirkonyum oksit (ZrO_2) ve karışımının nanoparçacıkları ile takviyenin polimetilmetakrilat reçinesinin eğilme mukavemeti üzerindeki etkisini değerlendirmek için yapılmıştır. Gereç ve yöntemler: 65 mm × 10 mm × 2.5 mm ölçülerinde toplam 60 akrilik reçine numunesi üretilmiştir. Örnekler her grupta on beş örneğe [kontrol grubu (C), ağırlıkça %3 PEEK grubu (P), ağırlıkça %3 zirkonya grubu (Z) ve ağırlıkça %1.5 PEEK ve ağırlıkça %1.5 ZrO_2 grubu (P-Z) hibrit grup.]. Numunelerin eğilme mukavemeti, evrensel test makinesinde üç nokta eğme testi kullanılarak değerlendirildi. İstatistiksel analiz, tek yönlü varyans analizi (ANOVA) kullanılarak yapıldı ve gruplar arası karşılaştırma, Tukey'nin post hoc analizi kullanılarak yapıldı. Bulgular: En yüksek ortalama eğilme mukavemeti grup P-Z'de (98.73 MPa), ardından grup P'de (86.22 MPa) ve grup Z'de (84.48 MPa) bulundu. En az ortalama eğilme mukavemeti kontrol grubundaydı (74.86MPa). Tek yönlü ANOVA, gruplar arasında anlamlı ($P<0.01$) bir fark ortaya koydu. Gruplar arasında ikili karşılaştırma, anlamlı bir farkın bulunmadığı P ve Z grupları dışında ($P=0.406$) tüm gruplar arasında anlamlı bir fark gösterdi ($P<0.05$). Sonuç: Hibrit takviyeli PEEK ve zirkonya, protez kaidesi reçinesi için etkili bir takviye materyali olarak kullanılabilir. Hibrit PEEK ve zirkonya ile güçlendirilmiş reçine, yüksek oklüzal kuvvetleri olan hastalarda ve daha önce çoklu protez kırığı deneyimi olan hastalarda alternatif bir tedavi seçeneği olabilir. Anahtar Kelimeler: eğilme mukavemeti, nanopartiküller, polimetilmetakrilat, polietereterketon, zirkonya

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Informed Consent: Not required.

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