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# CHARACTERISTICS ANALYSIS OF SEALANTS RESINS MATERIALS WITH NANOMETRIC PARAMETERS AFM AND SEM

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The degree of finishing of a surface depends on the structural features of the materials used but, for a good restoration, the material sealing ability is also a very important criteria. Thus we proposed the assessment *in vitro* of the topography, roughness and characteristics interface between material and teeth for three sealing and flow resin composite materials. The materials used were: Fotoseal (Remed Prodimpex), Permaseal (Ultradent Products Inc.) and Nanofill X flow (Schultzer RL). Topography and roughness were analyzed by Atomic Force Microscopy (Park SYSTEMS XE -100) for a 5μm × 5μm with scan rate of 0.5 Hz and a resolution of 256 × 256 pixels. Tridimensional topographic analysis was performed by analysis software (XEI - Image-Processing and Analysis). The microstructure and the interface enamel-sealant were analyzed by Scanning Electron Microscopy (JEOLJSM 6390ª Japan). There were differences in surface roughness among the sealants but it was no difference between the penetrations of material into enamel.

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*Keywords:* Sealants, Atomic force microscopy, Scanning electron microscopy

## 1. Introduction

Considering guidelines of World Health Organization which state that in 2025 DMFT index value to be 1, primary prevention measures are widely applied worldwide. The fluoridation, nutrition, dental hygiene, sealing of the teeth is a method of prevention of dental caries very efficiency at the pits and fissures. Sealing is indicated immediately after teeth eruption. The main purpose of sealing is closing retentive tooth reliefs and ecological niches of plaque stagnation.

Nowadays, the diversity of sealing materials and thus the choice of a material or another depends by many factors. An important parameter of choosing a material is its durability over time. The adherence of dental plaque to a surface is correlated with the degree of roughness of the surface material. The degree of roughness is higher so plaque retention is higher [1, 2].The wear resistance of a material is strongly related to structure of the material used and with the degree of surface roughness. Also, if the surface is smooth the wear resistance is higher. A smooth surface is fundamental for improving the longevity of the integrity for the teeth. In another case, the retention of dental plaque is higher and is favorable for development the decay.

The characteristics of the morphology of the material were systematically investigated by SEM, AFM, Optical Microscopy (MO), Energy-dispersive X-ray spectroscopy (EDS) and other different methods [1-7].

Also, there are the studies witch demonstrated that after surface sealant was applied, the micro-defect on the surface of composite resin decreased remarkably [8]. Also, the application of surface sealant could improve the surface quality and success of composite restorations [9].

Ethical and moral principles according to which operate in practice recommend the use of materials according to: their characteristics, the main indication and surface topography. The application of a technique or another by primary prevention depends very much by knowledge of

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the dentist in the domain. Therefore the study of the morphological characteristics is of maximal importance. The null hypothesized of this study is that for sealant materials there isn't difference between the roughness surfaces. The aim of this *in vitro* study was to evaluate the roughness, the topography and the interface between enamel and different sealing materials by AFM and SEM.

The objectives were: The analysis of surface morphology and roughness of sealing materials by AFM and the analysis of the microstructure and of the interface enamel-sealant by SEM.

## 2. Experimental

The materials used in this study are listed in the table 1.

*Table 1. Lists of investigated materials*

| Code | Brand Name | Manufacturer | Compositions | Batch | SM |
|---|---|---|---|---|---|
| F | Fotoseal | Remed Prodimpex | Monomers Dimethacrylate<br>Alkaline fluorides, oxides of titanium and silicon. | LOT 2/21.03.2012<br>CE 1868 | L |
| D | Permaseal | Ultradent Products Inc. | Bis GMAresin60%<br>Triethylene Glycol Dimethacrylate 40%<br>2-dimethylaminoethyl Methacrylate,<3% | ISO 9002<br>US PAT 75 | L |
| P | Nanofill X flow | Schultzer RL Superior GmbH, Hamburg/Germany | BIS-GMA-resin.<br>Filler content: 69 % (wt), 56 % (vol), filler particules<1,0 µm. | LOT 5106726<br>A3 | L |
| | SM - Setting Mechanism, L- Light curing | | | | |

### *2.1. The analysis of surface morphology and roughness of sealing materials by AFM*

The analysis of surface roughness, by AFM was made on the samples of three different sealants materials according to the table 1.

The materials were applied in conformity with manufacturer indication between two matrix strips of celluloid (Nr. 437 Alfred Becht GmbH-D 7600 Offenburg, Germany) and two glasses plates to obtain a flat surface. The source for polymerization was a conventional quartz halogen lamp (QTH), power density 570mW/cm$^2$ (3MESPE). The roughness and the topography of the surface were characterized by AFM (Park SYSTEMS XE -100) which is an important tool for imaging the materials surface in order to obtain information concerning the local surface. Its lateral resolution reaches 10 nm with scan sizes up to 100 µm. The AFM consists of a cantilever with a sharp tip (probe) at its end that is used to scan the specimen surface. The cantilever is typically silicon or silicon nitride with a tip radius of curvature on the order of nanometers. When the tip is brought into proximity of a sample surface, forces between the tip and the sample lead to a deflection of the cantilever. AFM was used in non-contact mode using single crystal silicon tip (with nominal radius < 10 nm), which was connected to a fixed substrate on a canti-lever.

The images were recorded with a scan rate of 0.5 Hz and a resolution of 256 × 256 pixels. For each specimen, two scans were carried out at each specimen surface quadrant at a scanning area of 5 µm × 5 µm. [3, 4 and 10].

### *2.2 The analysis of the microstructure and the interface enamel-sealant by SEM*

The microstructure and the interface enamel-sealant were analyzed by SEM (Scanning Electron Microscopy - JEOLJSM 6390$^a$ Japan). The study was conducted in vitro on human premolar and molars extracted for orthodontic or periodontal reasons, after obtaining informed consent of patients. The samples were divided randomly into 3 groups (GR) of equal length and

are sealed as follows.

- GR.1= Fotoseal (Remed Prodimpex);
- GR.2= Permaseal (Ultradent Products Inc.);
- GR.3 = Nanofill X flow (Schultzer).

The teeth were sealed using adhesive system consists of 3M™ Schotchbond Etch, Meta Bond 2 (Metabiomed). Sealing was performed in accordance with manufacturer's instructions. The samples were photo activated with halogen lamp (3M), stored in a physiological serum (48h), sectioned mesial-distal in the longitudinal direction (diamond discs) finished and conditioned with orthophosphoric acid with 37%for 5 seconds. The analysis of the interface between enamel and sealing materials was made by SEM. The measurement size of the hybrid layer was realized in three points for each side of each tooth, obtaining 12 measurements for each material [11, 12].

## 3. Results

Evaluation of the roughness revealed that the degree of roughness varies depending on the area of study. Permaseal material (Ultradent Products Inc.) presented the highest degree of roughness for a range of 5µm (Fig.1) compared with the other samples (Fig.2, 3), tab.2.

*Table 2. Analysis by AFM for the surface roughness Ra [nm] for 5 µm.*

| Material | µm | *Ra* [nm] | Rz(nm) |
|---|---|---|---|
| F | 5 | 4,689 | 58,59 |
| P | 5 | 14,74 | 157,89 |
| N | 5 | 6,45 | 75,807 |

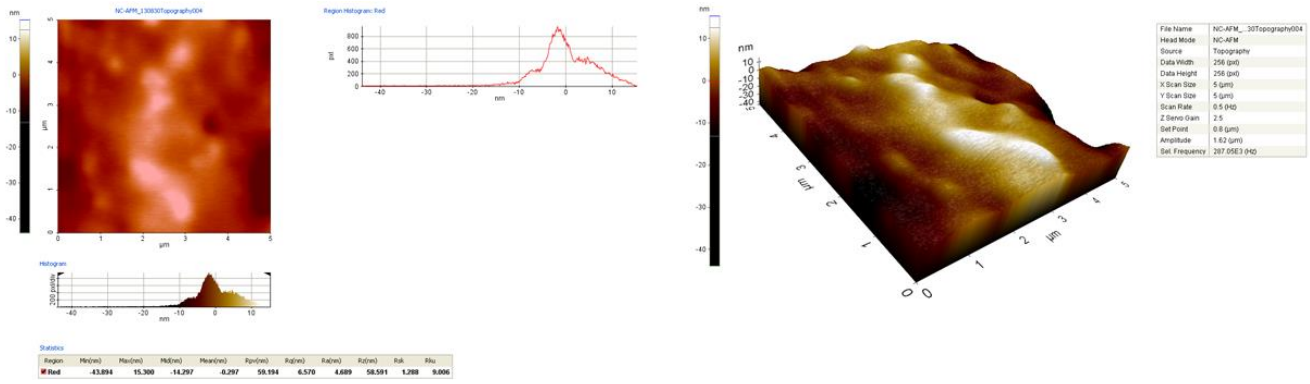


*Fig. 1-Atomic Force Microscopy for the sealant resin composite Fotoseal (REMED PRODIMPEX - RO), for 5 µm*

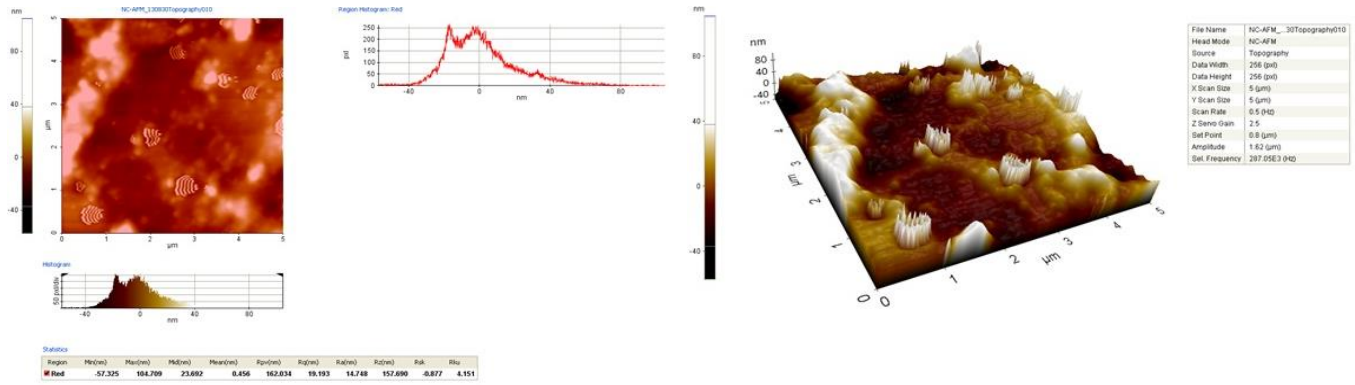


*Fig. 2-Atomic Force Microscopy for the sealant resin composite Permaseal (Ultradent), 5 µm*

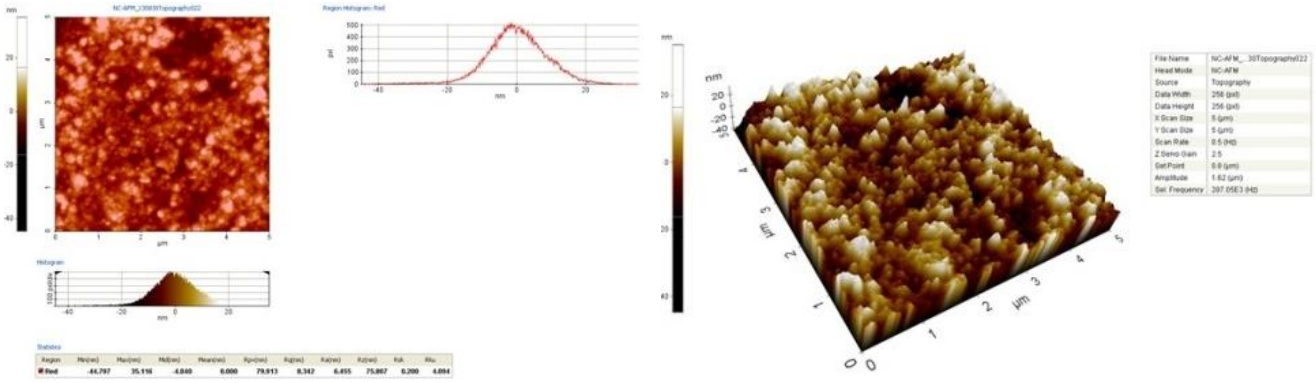
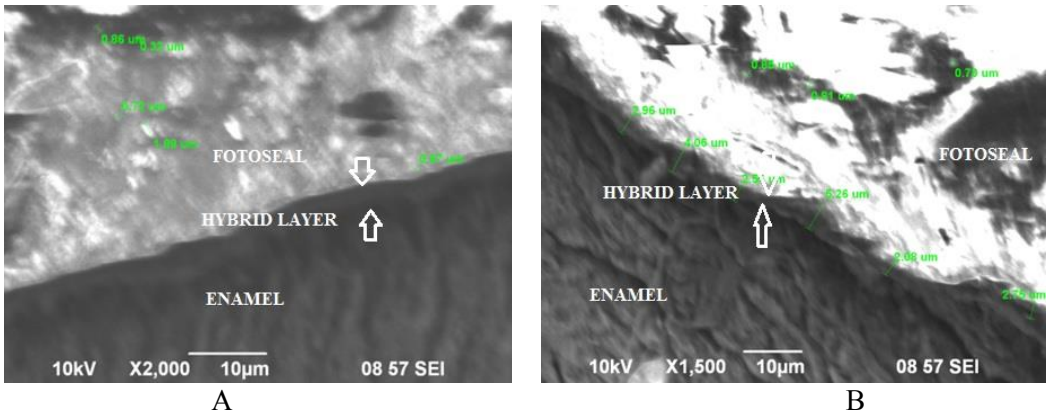


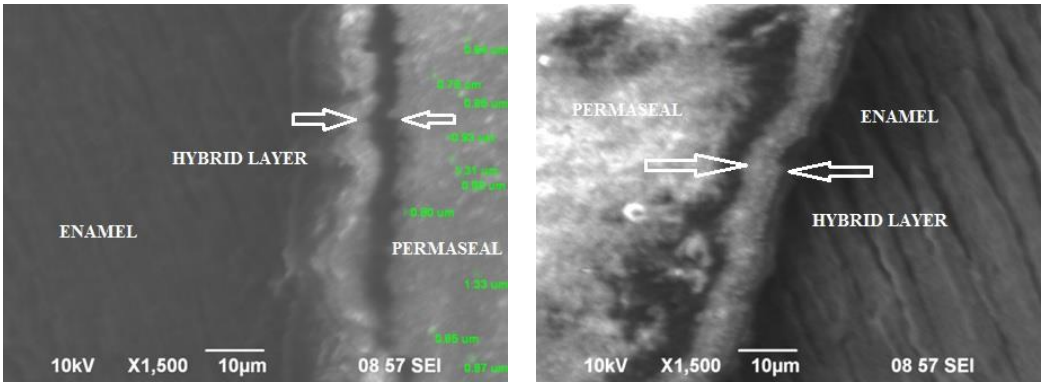
*Fig. 3- Atomic Force Microscopy for the sealant resin composite Nanofill X flow (Schultzer), 5 µm*

The SEM analysis indicates that there are differences between the shape, diameter, and the degree of filling of materials analyzed Fig.4 - 6.



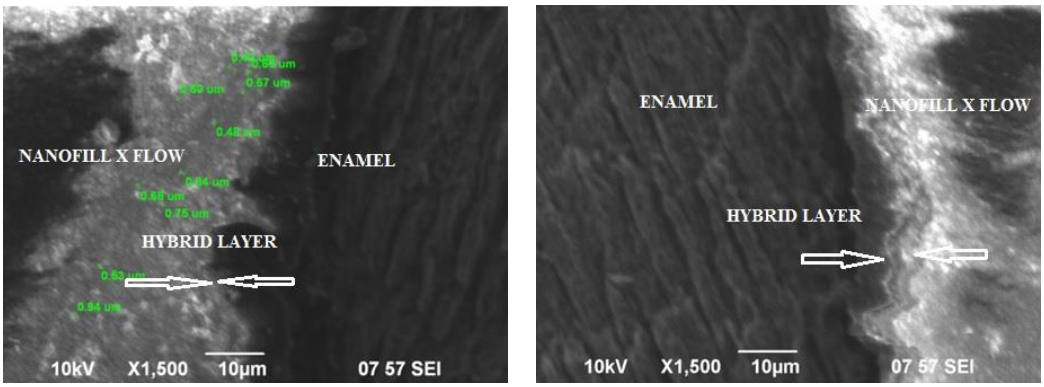

A B

*Fig.4–A and B - SEM micrograph of micro particle morphology and interface of enamel – sealing material Fotoseal (REMED PRODIMPEX - RO)*




A B

*Fig. 5 –A and B - SEM micrograph of micro particle morphology and interface of enamel – sealing material Permaseal (Ultradent)*




A B

*Fig. 6 - A and B - SEM micrograph of micro particle morphology and interface of enamel – sealing material Nanofill X flow (Schultzer)*

The analysis of the interface indicated that the sealing materials adhere to the enamel substrate and there is no significant differences between those (table no.3 and 4).

*Table 3 Dimension of the hybrid layer*

| | N | Minimum | Maximum | Mean | Std. Deviation |
|---|---|---|---|---|---|
| Fotoseal | 12 | 1,63 | 5,38 | 3,3692 | 1,02032 |
| Permaseal | 12 | 2,00 | 5,61 | 3,4225 | 1,19045 |
| Nanofil X flow | 12 | 2,14 | 4,61 | 3,1367 | ,76464 |
| Valid N (listwise) | 12 | | | | |

*Table 4 Correlations between the dimensions of the hybrid layer*

| | | Fotoseal | Permaseal | Nanofil |
|---|---|---|---|---|
| Fotoseal | Pearson Correlation | 1 | ,456 | ,461 |
| | Sig. (2-tailed) | | ,136 | ,132 |
| | N | 12 | 12 | 12 |
| Permaseal | Pearson Correlation | ,456 | 1 | ,336 |
| | Sig. (2-tailed) | ,136 | | ,286 |
| | N | 12 | 12 | 12 |
| Nanofil X flow | Pearson Correlation | ,461 | ,336 | 1 |
| | Sig. (2-tailed) | ,132 | ,286 | |
| | N | 12 | 12 | 12 |

*** Correlation is significant at the 0.01 level (2-tailed).*
** Correlation is significant at the 0.05 level (2-tailed).*

## 4. Discussions

The morphological characteristics of sealing materials influence their sustainability over time. Such a rougher surface material will favor the retention of plaque and thus will favor the creation of ecological niches that will maintain high levels of bacterial colonies. Depending on the conditions offered by the patient's oral environment this can cause carious lesions. Thus the use of effective means of dental prevention can be changed into a favorable means of tooth decay. The use of sealants material so that primary prevention and secondary prevention material in dental restorations seal is favorable for preserving tooth structure and restorative material respectively. Using a sealing material or another is influenced by its ability adhesion, wear resistance.

Fotoseal is a material that contains dimethacrylate monomers, alkaline fluoride, titanium and silicon oxides. Permaseal contain Bis-GMA and Nanofill - bisphenol glycidyl methacrylate - based monomer resin composite shows a high viscosity, high molecular weight that shows disadvantages in terms of substrate wettability. Instead, materials based on Bis-GMA shows a polymerization shrinkage reduced, which prevents dehiscence sealant substrate, especially when using an adhesive system performance with high adhesion capacity of 25 to 30 megapascals, resistant microleakage and nanoleakage. This is an advantage. Also, sealants which contain Bis-GMA shows a smaller internal conversion rate compared to those based UEDMA. The latter shows high molecular weights have an optimum viscosity (11,000 MPa at $23^0$C) and flexible urethane linkages that enhance endurance.

Atomic force microscopes (AFM) are extremely high-resolution scanning probe microscopes, with demonstrated resolution of fractions of a nanometer. The difference between the values obtained can be assigned the loading degree of materials with inorganic particles, and particle size components.

The oral environment is one of the factors that determine the clinical longevity of the materials. [13]. In clinical studies dental sealant Fotoseal ® has an immediate retention by 100% and after 3 years the average retention was 73.97% [14]. Thus considering the low roughness and penetrating ability of the enamel can be considered an optimal solution.

PermaSeal composite sealer is a light-cured, methacrylate-based, unfilled resin. It's low viscosity allows excellent penetration, and the ultrathin layer minimizes the need for occlusal adjustment. High integration ability of this material to the enamel was very good. This ensures the enamel surface reinforcement, and this is the main purpose of its use [15].

NANOFILL X FLOW is based on BIS-GMA-resin and inorganic filler particles <1, 0 μm. Filler content: 69 % (wt.), 56 % (vol). Low shrink low abrasion flowable light cure nano-composite with improved mechanical properties

Sealing materials used influence penetration in dentinal substrate. Possible causes for these differences are surprised sealing material flowing through the immediate application of the

source of large contraction after photo activation or due to polymerize. Inorganic filler particles function as true barriers to internal stress, so do not allow the emergence of a global tension, and after polymerization shrinkage is reduced [16]. This theory leads us to conclude that the sealant which. The composite resin without fillers has high polymerization shrinkage due to internal tensions accumulations thus determining the hybrid layer thickness reduction.

The two investigated composite materials had similar results regarding the interface quality and the marginal adaptation in dentin. The conventional resin cement associated with the adhesive system etch & rinse gave the lowest micro leakage values and formed the most homogeneous interface between the composite inlay and dental structure [17].

The analysis of the hybrid layer studied in two groups revealed no significant differences statistically Fotoseal 3.36 μm (± 1.02), Permaseal 3.45 μm (± 1.19) 3.13 μm flow Nanofil X (± 076).Similar studies show the sealant penetration deeper [18, 19].

## 5. Conclusions

Although there are differences in terms of material composition analysis, the penetration of materials into enamel was about equal, so the indication in the limits of this study is oriented toward the material with a lesser roughness, thus favoring dental self-cleaning.

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## References

[1] P.Khalichi&J.Singh, Biomaterials **30**, 452 (2008).
[2] E.M. Oong, S.O. Griffin, W.G. Kohn, B.F. Gooch, P.W. Caufield. JADA, **139**(3), 271 (2008).
[3] C.I. Saveanu&O. Dragos IJAR, **5**(3), 562 (2015).
[4] C.I. Săveanu *Plastic materials used in the caries prevention of dental caries dental prevention. Morpho-functional characteristics.* Press Gr.T.Popa, Iasi (2011).
[5] S.K. Gray&S.O. Griffin, JADA, **140**(1), 38(2009).
[6] A. Agrawal&A., Shigli, JISPPD, **30**(1), 51 (2012).
[7] P. H. Dos Santosa, S. Pavana, T. Y. U. Suzukia, A. L. F. Brisob, W. G. Assunçãoa, M. A. C. Sinhoretic & S.Consanic J Mech Behav Biomed Mater **4**, 433 (2011).
[8] H. JANG presented at the 86th General Session IADR congress, nr.0981 (2008).
[9] C. Anderson, A. L. F. Briso, R. H. Sundfeld,; P. H. Dos Santos JERD **22**(5), 324 (2010).
[10] C.I. Saveanu, O. Dragos, H. Chiriac JOAM, **14**(7-8), 826 (2012).
[11] C.I. Saveanu& O. Dragos DJNB, **7**(3), 1473 (2012).
[12] C.I. Saveanu& O. Dragos RJBL, **7**(6),7737 (2012).
[13] J.R. Heath & J.H. Wilson J. Oral. Rehabil. **3**, 121 (1976).
[14] A. Muntean presented at the Scientific Workshop Biological Adhesives: from Biology to Biomimetics Cluj Napoca, Romania, COST Action TD0906 WG3 & WG4, 9th -11th of April 2013 http://www.cost-bioadhesives.org.
[15] M., Anagnostou, G. Mountouris, N. Silikas, D. Kletsas, G. Eliades Dent Mater S0109-5641(15) (2015)
[16] M. Romănu, D. Bratu, S. Lakatos, Z. Floriţa, Polymerization in dentistry, Press Brumar, Timişoara (2000).
[17] Soanca, M.Rominu, M. Moldovan, C.I. Bondor, C. Nicola, A. Roman, D. J. N. B., **6**(1), 349 (2011).
[18] Nakabayashi N, Pashley DH Tokyo, Hybridization of dental hard tissue Press Quintessence International, 1998.
[19] C. I. Saveanu, A. Todiraşcu, I. Maftei, I. Danila RJOR **3**(4), 84 (2011).