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## Comparative Evaluation of Ultrasonically Activated Solvents on the Dissolution of a Bioceramic Root Canal Sealer: An In Vitro Study

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### ABSTRACT

**Aim:** To compare the efficacy of ultrasonic-activated solvents - xylene, orange oil, and Endosolv in dissolving a bioceramic root canal sealer under different immersion periods.

**Materials & methods:** Twenty standardized specimens of bioceramic sealer (BioActive RCS, SafeEndo Dental, Mumbai, India) were prepared using stainless-steel molds (6 mm × 4 mm). The specimens were weighed using an analytical balance and divided into three experimental groups (xylene, orange oil, and Endosolv; n = 5 each) and one control group (saline; n = 5). Each solvent group was further subdivided according to immersion time (2 and 5 minutes). Ultrasonic activation was performed for the corresponding durations, with solvent renewed each minute. After treatment, specimens were reweighted. The percentage weight loss was calculated. Data were analyzed using the Kruskal-Wallis test followed by Bonferroni post-hoc comparisons with significance set at p < 0.05.

**Result:** Xylene produced highest mean weight loss values among all solvents (p < 0.05). Ultrasonic activation significantly increased dissolution of bioceramic sealer compared to static immersion. The amount of dissolution increased with prolonged exposure time from 2 to 5 minutes.

**Conclusion:** Xylene demonstrated the greatest solvent efficacy for dissolving bioceramic sealer, especially when combined with ultrasonic activation. The dissolution potential of all solvents improved under ultrasonic agitation and with increased immersion duration

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residual infection and restore periapical health [2]. A key factor determining the success of retreatment is the complete removal of previous endodontic filling materials, which permits effective canal disinfection and optimal adaptation of new obturation materials. Several mechanical, thermal, and chemical techniques have been developed to aid in this process [3]. However, none can ensure complete removal of filling remnants from the root canal walls [4,5].

To improve cleaning efficiency, Passive Ultrasonic Activation (PUA) has been proposed as a useful adjunct. This method relies on ultrasonic energy to create acoustic streaming and cavitation within the solvent, enhancing its penetration and dissolving action without the need for continuous irrigation [6,7]. Such agitation has been shown to increase the

### INTRODUCTION:

Endodontic treatment can fail when root canal cleaning or obturation is incomplete. It allows necrotic debris and microorganisms to persist within the canal system and sustain periapical inflammation [1]. In such cases, non-surgical retreatment is preferred approach to eliminate

cleaning efficiency and debridement of the canal system [8]. When used in combination with appropriate solvents, PUA may enhance the dissolution of root filling materials during retreatment [7]. Nevertheless, its performance against bioceramic sealers has not been extensively studied.

Conventional sealers such as zinc oxide–eugenol, calcium hydroxide, and epoxy resin–based formulations adhere to dentin through both mechanical interlocking and chemical bonding [9]. In contrast, calcium silicate based bioceramic sealers have become popular due to their high biocompatibility, dimensional stability, and strong sealing ability [10,11]. Their alkaline nature and low cytotoxicity make them biologically favorable [12,13]. These same properties—especially their strong dentin adhesion—make them more resistant to removal during retreatment procedures [14].

Although solvents like chloroform and xylene are highly effective for softening conventional sealers, their potential health hazards, including toxicity and carcinogenicity, have encouraged the exploration of safer, biocompatible substitutes. Natural agents such as refined orange oil and other compounds including tetrachloroethylene and ethyl acetate have shown promise as less harmful alternatives [15]. Because sealers vary widely in their composition and physical properties, the ability of solvents to dissolve them may differ considerably [16].

Accordingly, the present in-vitro study aimed to evaluate and compare the efficiency of xylene, orange oil, and EndoSolv in dissolving a bioceramic root canal sealer under ultrasonic activation at two different immersion times.

## MATERIALS AND METHODS:

This in vitro experimental study was conducted using standardized stainless-steel molds in order to evaluate the effect of different solvents and ultrasonic activation periods on the dissolution of bioceramic root canal sealer.

**Sample preparation:** Twenty standardized specimens were prepared using stainless-steel molds (6 mm internal diameter  $\times$  4 mm height). A premixed, injectable calcium silicate–based sealer (BioActive RCS, SafeEndo Dental, Mumbai, India) was extruded directly into the molds placed on a clean glass slab to prevent air entrapment (Fig. 1a). A microscope slide lined with a thin cellophane strip was pressed gently on the surface to obtain a smooth, level finish. The filled molds were kept at 37 °C and 80 % relative humidity for 48 hours to ensure complete setting and standardized hydration

of the material across all samples.

After setting, the sealer specimens were carefully removed from the molds and trimmed with a scalpel to eliminate surface irregularities or flash (Fig. 1b). Each specimen was weighed three times using a digital analytical balance (Saffron, India; accuracy  $\pm$  0.001 g) (Fig. 1c), and the mean value was recorded as the baseline weight ( $W_0$ ). Specimens displaying visible voids, cracks, or surface defects were excluded from analysis.

**Grouping and intervention:** The specimens were randomly assigned (computer-generated sequence) into four solvent groups ( $n = 5$  each): xylene, orange oil (Biodinâmica, Ibirapuã, Brazil), EndoSolv E (Septodont, France), and normal saline (0.9 % NaCl) as the control. Each group was further divided into two subgroups based on immersion time—2 minutes and 5 minutes (Table 1). Each specimen was placed individually in a glass vial containing 1 mL of freshly prepared solvent at 23  $\pm$  1 °C (Fig. 1d). Ultrasonic activation was carried out in a bench-top ultrasonic bath (S.S. Ultrasonic Cleaner, Vishal Scientifics, India) operating at a frequency of 30 kHz and output power of 100 W (Fig. 1e). The vials were arranged within the water-filled chamber of the bath to ensure uniform energy distribution. Ultrasonic agitation was applied for 30 seconds at full power during both immersion intervals (2 and 5 minutes). The setup ensured indirect transmission of ultrasonic energy through the vial wall, avoiding direct mechanical contact with the sealer specimens.

For the passive (non-activated) subgroups, samples were immersed in the respective solvents at room temperature for the same time periods without ultrasonic exposure. After treatment, specimens were rinsed with 10 mL of distilled water, blotted dry with absorbent paper, and placed in a dehumidifier at 37  $\pm$  1 °C for 24 hours. Drying was continued until two consecutive weight readings differed by less than 0.001 g. The final stabilized mass was recorded as  $W_t$ , obtained from the mean of three independent measurements.

**Evaluation and statistical analysis:** The percentage weight loss of each specimen was calculated using formula: Weight loss (%) =  $(W_0 - W_t)/W_0 \times 100$ ; where  $W_0$  is the initial weight and  $W_t$  is the final weight after immersion. Mean dissolution percentages and standard deviations were computed for each group and immersion period. Data analysis was performed using STATA 12.0 software (StataCorp, College Station, TX, USA). The normality of data distribution was verified prior to inferential testing. Intergroup

comparisons were made using one-way analysis of variance (ANOVA) followed by Tukey's post-hoc test, while paired t-tests were employed for intragroup comparisons across immersion times. A p-value < 0.05 was considered statistically significant.

## RESULTS:

This in vitro study compared the dissolution efficacy of xylene, orange oil, and EndoSolv on bioceramic sealer (BioActive RCS, SafeEndo Dental, Mumbai, India) under ultrasonic activation. The mean and standard deviation values for pre- and post-treatment specimen weights are shown in Table 2.

One-way ANOVA (Table 2) indicated statistically significant difference among the pre-treatment weights of the groups ( $p = 0.024$ ). Post-treatment values showed no significant difference ( $p = 0.273$ ). However, the difference between pre- and post-treatment weights revealed a highly significant variation among the solvents ( $p = 0.001$ ).

Paired t-test analysis (Table 3) demonstrated that all groups showed measurable weight reduction. Xylene produced the greatest mean weight loss ( $0.0062 \pm 0.000837$  g;  $p < 0.001$ ). This was followed by EndoSolv ( $0.0018 \pm 0.000837$  g;  $p = 0.009$ ) and orange oil ( $0.0016 \pm 0.000548$  g;  $p = 0.003$ ). The control group also demonstrated a small but statistically significant weight change ( $0.0016 \pm 0.000548$  g;  $p = 0.003$ ).

Post hoc Tukey comparisons (Supplementary Table S1) confirmed that xylene resulted in significantly greater weight loss compared with both orange oil (mean diff = 0.0046,  $p < 0.001$ ) and EndoSolv (mean diff = 0.0044,  $p < 0.001$ ). The difference between orange oil and EndoSolv was not significant ( $p = 1.000$ ). These results indicate statistically significant variation in sealer dissolution among the tested solvents with xylene showing the highest mean weight loss (Figure 2).

All solvents exhibited a time-dependent increase in dissolution, with higher mean weight loss observed after 5 minutes compared with 2 minutes (Table 4). The difference was most pronounced in xylene subgroup.

## DISCUSSION:

This study assessed the dissolution efficiency of xylene, orange oil, and EndoSolv on a premixed bioceramic root canal sealer (BioActive RCS, SafeEndo Dental, Mumbai, India) when subjected to ultrasonic activation. The results showed that xylene had the strongest dissolving effect, followed by EndoSolv and orange oil, while saline (control)

produced minimal changes. The small weight loss seen in the control samples can be attributed mainly to ultrasonic agitation rather than chemical dissolution. This indicates that solvent composition remains the primary determinant of solubility, with ultrasonic energy and immersion time acting as reinforcing factors.

The superior performance of xylene aligns with previous investigations such as those by Aiswarya et al. (2023) and Martos et al. (2006)<sup>[3,8]</sup>, which demonstrated its high capacity to soften or dissolve epoxy resin- and MTA-based sealers. Xylene is non-polar molecular structure allows it to penetrate and disrupt hydrophobic polymer chains within sealers. This leads to disintegration and detachment. In contrast, orange oil and EndoSolv possess limited compatibility with the polar calcium silicate matrix of bioceramic sealers, explaining their relatively weaker dissolution. Their comparable performance suggests that the limitation is primarily chemical rather than procedural. Orange oil is valued for its low toxicity and tissue tolerance but is chemically less aggressive than xylene<sup>[8]</sup>. Clinically, this makes it a suitable choice in retreatment situations where patient safety takes precedence over maximal dissolving power. EndoSolv, although designed for resin-based materials, demonstrated moderate efficacy, which may be attributed to its solvent base being less reactive toward inorganic bioceramic constituents.

Ultrasonic activation significantly improved the dissolution capacity of all solvents. Passive Ultrasonic Activation (PUA) enhances fluid movement through cavitation and acoustic microstreaming, increasing the solvent's ability to penetrate and interact with the sealer surface. This synergistic effect between chemical and mechanical energy is supported by Cavenago et al. (2014)<sup>[6]</sup>, who reported superior removal of filling materials with ultrasonically energized solvents. Similarly, increasing immersion time led to greater material loss across all groups, corroborating the observations of Mushtaq et al. (2012)<sup>[16]</sup> that prolonged solvent exposure improves softening and dissolution efficiency.

The limited solubility of bioceramic sealers can be explained by their material chemistry. Zhang et al. (2015)<sup>[17]</sup> and Wang et al. (2014)<sup>[18]</sup> showed that calcium silicate sealers form a hydroxyapatite layer upon setting, which chemically bonds to dentin and makes them inherently resistant to solvent attack. This explains the relatively lower dissolution compared to traditional sealers. The differing solvent responses are also influenced by molecular polarity, viscosity, and volatility, as described by

Alzraikat et al. (2016) [19] and Whitworth and Boursin (2000) [20].

Temperature may further influence the solubility process. Bodrumlu et al. (2008) [21] observed that heating solvents can improve their dissolving power, suggesting that combining ultrasonic energy with mild heat might produce a stronger effect. Less toxic options such as limonene-based and polyethylene glycol-based solvents have also shown promise in reducing cytotoxicity while maintaining adequate softening ability (Wourms et al., 1990; Vajrabhaya et al., 2004) [22,23]. The enhanced molecular motion and localized cavitation caused by ultrasound can further increase these solvents' reactivity, as described by Weller et al. (1980) and Ordinola-Zapata et al. (2019) [8,24].

However, limitations exist. This study's in-vitro design cannot fully simulate clinical complexity of root canal environment. Canal curvature, dentinal irregularities and temperature fluctuations influence behavior of solvent and sealer retrieval in vivo. Wennberg and Orstavik (1989) and Kaplowitz (1991) [25, 26] emphasized that such anatomical challenges significantly affect retreatment outcomes. The specimens in our study were exposed to solvents on a flat surface. This allowed uniform sealer access but likely overestimated dissolution compared to clinical scenario in confined root canal system. The experimental design included a limited sample size and tested only three solvents (xylene, orange oil, and EndoSolv) at two immersion periods using a single ultrasonic activation setting. Dissolution was evaluated solely by physical quantity i.e., weight loss percentage. No SEM or FTIR was used to characterize surface changes or to confirm chemical degradation.

The clinical relevance of solvent toxicity also warrants attention. As Chutich et al. (1998) [27] reported, some potent solvents carry cytotoxic risks, making it essential to balance effectiveness with biocompatibility. Present study focused primarily on dissolving efficacy without evaluating biocompatibility or cytotoxic potential of solvents. This factor is crucial for clinical safety. Emerging materials such as resin-coated gutta-percha and newer MTA-based sealers have shown greater resistance to dissolution (Kulkarni et al., 2016; Oyama et al., 2002; Rawtiya et al., 2013) [28-30]. This underscores the need for continued innovation in solvent formulations and retreatment protocols. This paper does not assess sequential use of multiple solvents such as xylene followed by orange oil. Such strategies are important in real clinical cases.

Clearly, xylene demonstrated the highest dissolution capacity for bioceramic sealer when used with ultrasonic activation, whereas orange oil and EndoSolv showed moderate but comparable effects. Ultrasonic agitation clearly enhanced solvent action, though the solvent's intrinsic chemistry remained the dominant factor determining performance. Future research should aim to optimize activation parameters, evaluate solvent combinations, and identify safer alternatives that retain high dissolving efficiency for clinical endodontic retreatment.

#### Future Directions:

Future studies should evaluate action of solvent inside extracted teeth or canal models that mimic real clinical conditions. A wider range of newer, biocompatible solvents such as eucalyptol or limonene-based agents with large sample size should be investigated as safer alternatives to xylene [22,23]. The influence of different ultrasonic parameters such as power, frequency and activation time should be examined to determine optimal settings for maximum solvent efficiency. Sequential solvent applications could be explored to assess possible synergistic effects in dissolving bioceramic sealers. Advanced techniques like SEM or FTIR analysis should be used to study degradation of sealer more precisely. Evaluating the cytotoxicity and tissue compatibility of solvents is essential for clinical translation.

#### CONCLUSION:

Within the limitations of this in-vitro study, the following conclusions were drawn:

- Xylene demonstrated the highest dissolving efficacy against bioceramic sealer.
- Orange oil and EndoSolv exhibited moderate but comparable dissolving abilities.
- Ultrasonic activation enhanced the dissolution potential of all solvents.
- Prolonged immersion time increased solvent efficacy.

Therefore, xylene can be considered the most effective solvent for bioceramic sealer removal when used with ultrasonic activation. Clinical use should balance its effectiveness with safety considerations.

#### List of abbreviations used:

ANOVA	Analysis of Variance
FTIR	Fourier Transform Infrared Spectroscopy
MTA	Mineral Trioxide Aggregate
PUA	Passive Ultrasonic Activation
SD	Standard Deviation
SEM	Scanning Electron Microscopy

**FIGURE LEGENDS:**

**Graphical Abstract** – Visual summary of the in-vitro study comparing the dissolving efficacy of xylene, orange oil, and EndoSolv on bioceramic root canal sealer under ultrasonic activation.

**Figure 1. Preparation and weighing of bioceramic sealer specimens.** (a) Stainless-steel mold (6 mm × 4 mm) filled with freshly mixed bioceramic sealer to create uniform cylindrical specimens. (b) Set sealer cylinders after removal from molds with standardized dimensions and smooth surface finish prior to immersion testing. (c) Analytical balance used for pre- and post-immersion weighing. (d) Individual samples immersed in different solvents in labeled vials. (e) Bench-top ultrasonic bath used for solvent activation of bioceramic sealer specimens.

**Table 1. Experimental grouping of specimens.**

**Figure 2. Mean dissolution of bioceramic sealer specimens in different solvents.** Xylene showed the highest dissolution compared to orange oil,

EndoSolv, and control ( $p < 0.05$ ). Error bars represent standard deviation.

**Table 2. One-way ANOVA comparing mean pre- and post-treatment weights and differences among solvent groups.** Analysis of variance showing significant differences in pre-treatment weights ( $p = 0.024$ ) and pre-post weight differences ( $p = 0.001$ ), indicating variation in the dissolution capacity of tested solvents.

**Table 3. Paired t-test comparing pre- and post-treatment weights within each solvent group.** Mean weight loss ( $\pm$  standard deviation) before and after ultrasonic activation. All groups showed statistically significant differences ( $p < 0.05$ ), with xylene exhibiting the highest mean loss.

**Table 4. Effect of immersion time and solvent type on bioceramic sealer dissolution under ultrasonic activation.** All solvents exhibited increased dissolution at 5 minutes compared with 2 minutes, indicating a time-dependent effect.

**IMAGE FILES**

**GRAPHICAL ABSTRACT**

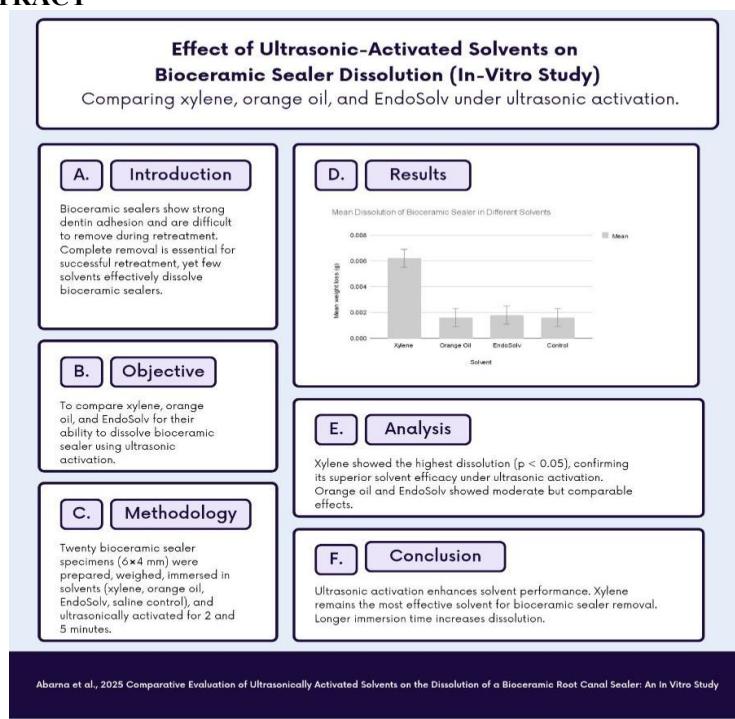


Fig. 1



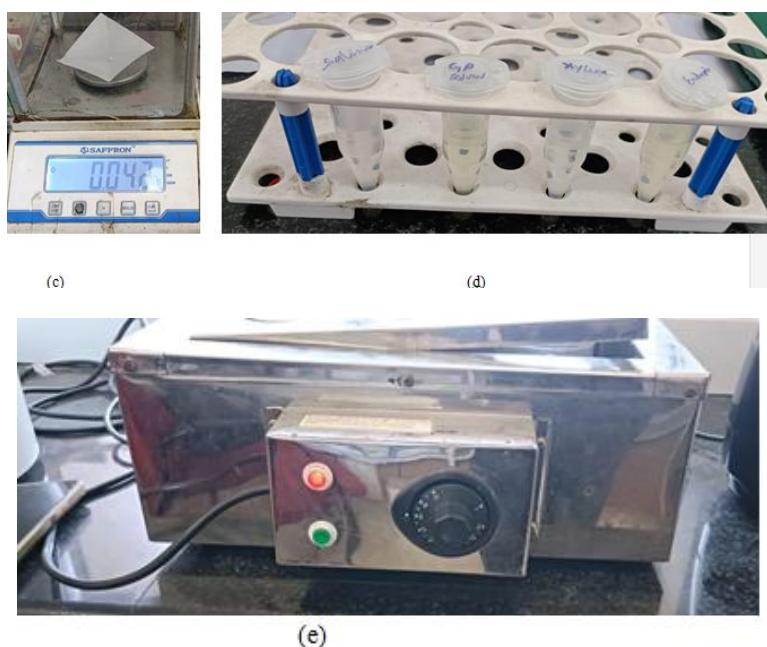


Table 1

Grp	Solvent used	Manufacturer	n	Activation mode	Immersion time (mins)
G1	Xylene	Analytical grade (Local supplier)	5	Passive and Ultrasonic	2 and 5
G2	Orange Oil	Biodinâmica, Ibiporã, Brazil	5	Passive and Ultrasonic	2 and 5
G3	EndoSolv	Septodont, France	5	Passive and Ultrasonic	2 and 5
G4	Normal Saline (Control)	Analytical grade (Local supplier)	5	Passive and Ultrasonic	2 and 5

Fig. 2

Mean Dissolution of Bioceramic Sealer in Different Solvents

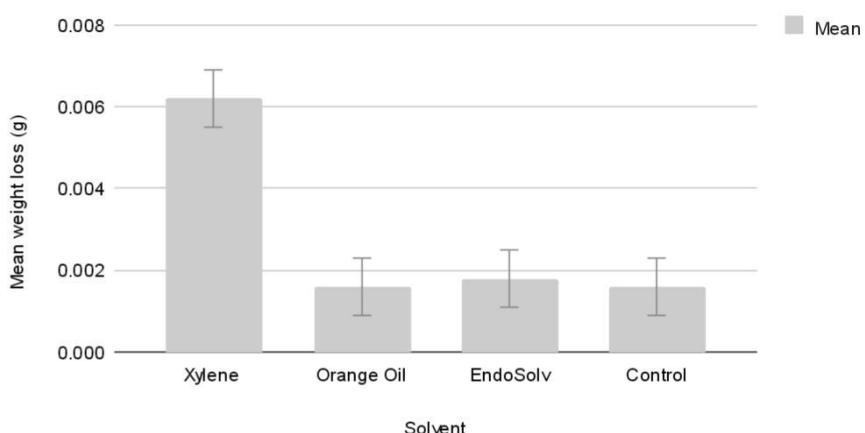


Table 2

		n	Mean	Std. Deviation	p-value
Pre	Xylene	5	0.04440	0.001140	0.024
	Orange Oil	5	0.03480	0.006760	
	EndoSolv	5	0.03780	0.004868	
	Control	5	0.03980	0.002864	
	Total	20	0.03920	0.005425	
Post	Xylene	5	0.03820	0.000837	0.273
	Orange Oil	5	0.03320	0.006648	
	EndoSolv	5	0.03600	0.005148	
	Control	5	0.03820	0.002775	
	Total	20	0.03640	0.004593	
Difference	Xylene	5	0.00620	0.000837	0.001

	Orange Oil	5	0.00160	0.000548	
	EndoSolv	5	0.00180	0.000837	
	Control	5	0.00160	0.000548	
	Total	20	0.00280	0.002118	

**Table 3**

		Mean Difference	Std. Deviation	Std. Error Mean	p-value
Xylene	Pre - post	0.006200	0.000837	0.000374	0.000
Orange Oil	Pre - post	0.001600	0.000548	0.000245	0.003
EndoSolv	Pre - post	0.001800	0.000837	0.000374	0.009
Control	Pre - post	0.001600	0.000548	0.000245	0.003

**Table 4**

Solvent	Immersion Time (mins)	Mean Weight Loss (g) ± Std. Deviation	p-value (within group)
Xylene	2	0.0051 ± 0.0006	< 0.001
	5	0.0062 ± 0.0008	< 0.001
Orange Oil	2	0.0012 ± 0.0004	0.005
	5	0.0016 ± 0.0005	0.003
EndoSolv	2	0.0014 ± 0.0007	0.012
	5	0.0018 ± 0.0008	0.009
Saline (control)	2	0.0013 ± 0.0004	0.017
	5	0.0016 ± 0.0005	0.003

**Supplementary Table S1. Post hoc Tukey pairwise comparison among solvent groups**  
Multiple comparison results identifying significant differences in dissolution between solvents. Xylene showed significantly greater weight loss compared

to orange oil and EndoSolv ( $p < 0.001$ ), while no significant difference was observed between orange oil and EndoSolv.

Dependant variable			Mean Difference (I-J)	Std. Error	Sig.
Pre	Xylene	Orange Oil	0.009600*	0.002809	0.021
		EndoSolv	0.006600	0.002809	0.192
		Control	0.004600	0.002809	0.726
	Orange Oil	Xylene	-0.009600*	0.002809	0.021
		EndoSolv	-0.003000	0.002809	1.000
		Control	-0.005000	0.002809	0.564
	EndoSolv	Xylene	-0.006600	0.002809	0.192
		Orange Oil	0.003000	0.002809	1.000
		Control	-0.002000	0.002809	1.000
Difference	Xylene	Orange Oil	0.004600*	0.000447	0.000
		EndoSolv	0.004400*	0.000447	0.000
		Control	0.004600*	0.000447	0.000
	Orange Oil	Xylene	-0.004600*	0.000447	0.000
		Orange Oil	-0.000200	0.000447	1.000
		Control	0.000000	0.000447	1.000
	EndoSolv	Xylene	-0.004400*	0.000447	0.000
		Orange Oil	0.000200	0.000447	1.000

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