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## Extraction of Citric Acid From Orange Peel as Whitening Ingredient of Toothpaste

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### ABSTRACT

Citrus is a fruit plant with a high production rate in Indonesia's plantation industry. A wide variety of content in citrus are beneficial for body, such as 40 mg of calcium per 100 mg of citrus are beneficial for teeth and bones. In addition, there is also Citric Acid compound. Citric acid can replace flouride and detergents that usually use as whitening ingredients of toothpaste to whiten teeth. The purpose of this research is to extract citric acid from orange peel as teeth whitener. This research is a quasi-experimental. Citric acid extraction was carried out on the type of Sweet Orange (*Citrus Aurantium L*), Tangerine (*Citrus Reticulata Blanco* or *Citrus Nobilis*), Pommelo (*Citrus Maxima Merr*, *Citrus Grandis Osbeck*), and Lemon (*Citrus Limon Linn*). Citric acid's ability test as teeth whitener was performed on premolar teeth with concentrations of 2,5%, 5%, and 10%. The experiments was replicated 3 times, and teeth were measured using *Shade Guide VITA Classical*. The result of this research showed that citric acid in every kind of orange peel with various concentration have different abilities on whitening teeth. The highest color level obtained from Tangerin peel's citric acid concentration of 5%.

**Keyword:** orange peel, citric acid, teeth whitener, whitening ingredient, extraction

### INTRODUCTION

Citrus is one of plant that produce very high number quantity at indonesian agriculture industry. (ditbuah, 2012) There are many ingredient inside citrus fruit that beneficial to the body, according to Perdue University Horticulture and Landscape Architecture in every 100 grams of citrus contains 40 mg of calcium which beneficial to bones and teeth. However with the exact amount, orange peel contained 161 mg calcium. Orange peel (*Citrus reticulata*) contain many compound like *Tangeraxanthin*, *Tangeritin*, *Terpinen-4-ol*, *Terpineolene*, *Tetradecanal*, *Threonine*, *Thymol*, *Thymyl-methyl-ether*, *Tryptophan*, *Tyrosine*, *Cis-3-hexenol*, *Cis-carveol*, *Citric-acid*, *Citronellal*, *Citronellic-acid*, *Citronellyl-acetate*, *Cystine*, *Decanal*, *Decanoic- acid*, *Decanol*, *Nobiletin*. (Pan et al, 2002) Citric acid compound known had cleansing effect which can replace flouride and detergent on toothpaste for whiten teeth. The purpose of these research is testing extract of citric acid as whitening compund in toothpaste.

### METHODS

#### Materials

Sweet Orange (*Citrus Aurantium L*), Tangerine (*Citrus Reticulata Blanco* or *Citrus Nobilis*), Pomelo (*Citrus Maxima Merr*, *Citrus Grandis Osbeck*), dan Lemon (*Citrus Limon Linn*). Testing citric acid potency as whitening compound to premolar teeth.

#### Extraction of Orange Peels

Separate orange peel from the fruit and cut into little pieces. Prepare oven with 50°C and insert these orange peels, wait until dried. Blend and filter orange peel that dried.

#### Observing Teeth Color

Observing teeth color done when before, after discoloration of the teeth and after soaking it with Orange peels citric acid. Observations were made subjectively using *Shade Guide Vita*

## Teeth Discoloration

Polish 36 pieces Premolar teeth with nail polish on root part until CEJ part 3 times. These teeth insert to discoloration fluid that made of 10gr robusta coffee and 180 ml water (boil until 100°C). Switch the fluid every 24 hours during 1 week.

## Orange Peels Citric Acid

Prepare orange peels extract each 2 grams, mix it with 75 distilled water and 25 ml methanol. Then stir it using tools stirrer magnetic for 30 minutes with 7th speed, and save it on volumetric flask. Next is preparing to the destillation phase for extracting acid citric on orange peel. Then take some the result from destillation for get active compound analyzed using GC-MS.

## Treatment Stage

Insert the discolored teeth into each flask that has orange peel extract. Place it on incubator (oven) with 50°C heat for 25 seconds, then lower the heat to 37°C for 30 minutes. These will produce whiten color to the teeth.

## GC-MS Analysis

Gas Chromatography – Mass Spectrometry (GC-MS) Analysis tested with these step :

1. For get the detail composition, then orange peel sample centrifuged is analysed with using GC-MS
2. Sample is centrifuged with 5000 rpm speed in 20 minutes
3. Take Supernatant (liquid) and placed on beaker glass
4. Hexan solution inserted on beaker glass with sample : hexan, 1 : 3 Ratio
5. Mix sample using magnetic stirrer in 30 minutes
6. Separeate sample and hexan using separatory funnel
7. Take hexan solution and analys it using GC-MS

## Statistical Analysis

Analyze and interpret data using IBM SPSS 20 software. In this study conducted two test, there are difference test of various types and concentrations of orange peel's extract to ability to whiten teeth using One Way Anova, and the second test is Paired T-test that used to analyze change level between discolored teeth and teeth after treatment.

## RESULT AND DISCUSSION

### GC-MS Test Result

Orange peel extract that have distilled tested using GC-MS method for seeing active compound that active inside. From GC-MS test giving these as result:

Table 1. GC-MS Test Result

Peak No	R. Time	Area %	Name
1	43.771	21.46	methyl dihydromalvalate
2	47.782	20.17	Hexadecanoic acid, 1-(hydroxymethyl)-1,2-ethanediyl ester
3	51.298	33.38	9-Octadecenal, (Z)
4	67.940	16.69	Cholest-5-en-3-ol (3.beta.), carbonochloridate
5	67.969	8.30	Androstan-3-one, 17-hydroxy-1,17-dimethyl-, (1.alpha.,5.alpha.,17.beta.)

The tabel above telling us that orange peel have several active compound, like one that peak no 2, there are *Hexadecanoic acid 1-(hydroxymethyl)-1,2-ethanediyl ester* that have cluster like these:

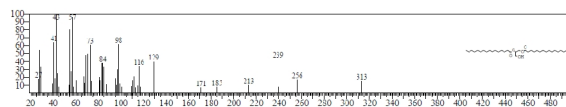


Figure 1. Mass Spectrum of *Hexadecanoic acid*

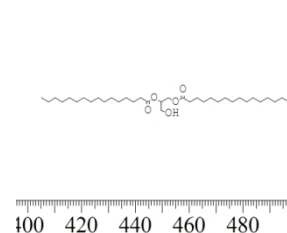


Figure 2. *Hexadecanoic acid* cluster

From chart above, Hexadecanoic acid have OH cluster that known can whiten teeth. in addition Hexadecanoic acid have potent as anti-bacterial and anti-fungi. Next on peak number 4 have active compound Cholest-5-en-3-ol or cholesterol that can functioned as emulsion stabilizer on toothpaste. On peak number 5, there are active compound Androstan which have OH cluster and can functioned as anti-inflammation on gum.

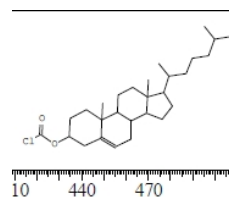


Figure 3. Cholest cluster

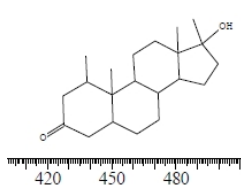


Figure 4. Androstan cluster

**Color Changing After Testing**

Color measurement test us using Shade Guide VITA Classical that have 16 category color. The color that inside shade guide start from most white to bright to most dark. The color that have sorted as their order, and the order score is :

B1=1, C1=2, A1=3, B2=4, D2=5, C2=6, B3=7, A2=8, B4=9, C3=10, A3=11, D3=12, D4=13, C4=14, A4=15, A35=16

and these the result of change discolored teeth that have soaked on extract acid citric orange peel as 30 minutes, with 4 kinds orange peel and 3 kinds differrent concentration.

Table 2. Result of Ability Test Citric Acid Orange Peel as Teeth Whitening at Various Concentrations

No	Citrus	Concen- tration	Dicolored Score	Dental Score after Treatment			Mean		
				I II III					
				I	II	III			
1	Sweet	2,5%	C4	14	6	6	6	6	
		5%			4	4	4		4
		10%			2	2	2		2
2	Tange- rine	2,5%	C4	14	4	2	7	4,33	
		5%			2	1	1		1,33
		10%			7	7	7		7
3	Pomelo	2,5%	C4	14	8	10	14	10,6	
		5%			10	10	7		9
		10%			7	10	10		9
4	Lemon	2,5%	C4	14	7	7	4	6	
		5%			10	10	7		9
		10%			4	4	4		4

From tabel above, we know that citric acid on tangerine peel with 5% concentration and sweet orange peel with 10% concentration, give result more better than the other.

**Data Analysis Result**

From One Way Anova test we get score Sig 0,000 (<0,05) which show different effect that significant between variation kinds and concentration orange peel. On the other side, Paired T-test also show sig 0,000 (<0,005) and prove that significant transformation between discolored teeth and soaked extract orange peel.

Table 3 Result of One Way Anova parametric statistical test

Source	df	Mean Square	F	Sig.
<b>Intergroup</b>	11	26,816	11,492	<b>0,000</b>

Table 4. Result of Paired T-test parametric statistical test

	Mean	T	df	Sig.
Pair 1 Discolored score – Score after soaking on citrus peel	7,972	15,105	35	<b>0,000</b>

**CONCLUSION**

Based on research which has been done, we get result that citric acid that contained on orange peel can whiten the teeth, futhermore these orange peel have active compund that function as anti-bacterial, anti-fungi, emulsion stabilizer and anti-inflamation on gum. for getting acid acid on orange peel that can function as teeth whitener can be done with extraction. after testing on discolored teeth, we have result that soaking tangerine peel with 5% concretion give the best white result than other orange peel and concentrat.

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