

BAB V

KESIMPULAN DAN SARAN

A. Kesimpulan

Berdasarkan dari hasil penelitian yang telah dilakukan dapat diperoleh kesimpulan bahwa:

Pertama, myrisetin dapat dibuat sediaan serum yang memenuhi uji mutu fisik sediaan meliputi uji organoleptis, pH, dan viskositas.

Kedua, formula 1, formula 2 dan formula 3 serum myrisetin tidak stabil secara fisik selama proses penyimpanan dengan berbagai variasi konsentrasi *sodium gluconate*.

B. Saran

Penelitian ini masih banyak kekurangan, maka perlu dilakukan penelitian lebih lanjut mengenai:

Pertama, perlu dilakukan uji DPPH sediaan serum myrisetin.

Kedua, perlu dilakukan uji difusi sel menggunakan menggunakan membran biologis untuk mengetahui serum myrisetin yang dapat berpenetrasi ke dalam kulit.

Ketiga, serum myrisetin dibuat dalam bentuk enkapsulasi.

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Lampiran 1. Sertifikat analisis myricetin.

TOCRIS
a biotechne brand

Print Date: Jul 20th 2017

Certificate of Analysis

www.tocris.com

Product Name: Myricetin

Catalog No.: 6189

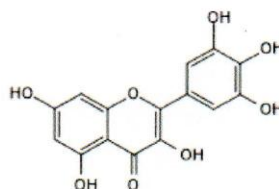
Batch No.: 1

CAS Number: 529-44-2

IUPAC Name: 3,5,7-Trihydroxy-2-(3,4,5-trihydroxyphenyl)-4H-1-benzopyran-4-one

1. PHYSICAL AND CHEMICAL PROPERTIES

Batch Molecular Formula: $C_{15}H_{10}O_8 \cdot H_2O$
Batch Molecular Weight: 336.26
Physical Appearance: Yellow solid
Solubility: DMSO to 100 mM
 ethanol to 50 mM
Storage: Store at -20°C
Batch Molecular Structure:



2. ANALYTICAL DATA

HPLC: Shows 97.7% purity
¹H NMR: Consistent with structure
Mass Spectrum: Consistent with structure
Microanalysis:

	Carbon	Hydrogen	Nitrogen
Theoretical	53.58	3.6	
Found	53.6	3.57	

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Product Information
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Product Name: Myricetin

Catalog No.: 6189

Batch No.: 1

CAS Number: 529-44-2

IUPAC Name: 3,5,7-Trihydroxy-2-(3,4,5-trihydroxyphenyl)-4H-1-benzopyran-4-one

Description:

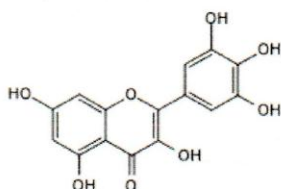
Irreversible TrxR inhibitor (IC_{50} = 0.62 μ M). Exhibits concentration-, time- and NADH-dependent TrxR inhibition. Results in the oxidation of Trx and reduced TrxR activity in vitro in addition to the accumulation of cells in sub-G₁ phase. Reduces neoplastic transformation and induces cell death in cancer cell lines. Chemotherapeutic.

Physical and Chemical Properties:
Batch Molecular Formula: C₁₅H₁₀O₈·H₂O

Batch Molecular Weight: 336.26

Physical Appearance: Yellow solid

Minimum Purity: >97%

Batch Molecular Structure:

Storage: Store at -20°C

CAUTION - This product is light sensitive and we recommend that the solid material and any solutions obtained are protected from exposure to light.

Solubility & Usage Info:

DMSO to 100 mM

ethanol to 50 mM

Stability and Solubility Advice:

Some solutions can be difficult to obtain and can be encouraged by rapid stirring, sonication or gentle warming (in a 45-60°C water bath).

Information concerning product stability, particularly in solution, has rarely been reported and in most cases we can only offer a general guide. Our standard recommendations are:

SOLIDS: Provided storage is as stated on the product label and the vial is kept tightly sealed, the product can be stored for up to 6 months from date of receipt.

SOLUTIONS: We recommend that stock solutions, once prepared, are stored aliquoted in tightly sealed vials at -20°C or below and used within 1 month. Wherever possible solutions should be made up and used on the same day.

References:

Devi et al (2015) Molecular mechanisms underlying anticancer effects of myricetin. *Life.Sci.* **142** 19. PMID: 26455550.

Lu & Holmgren et al (2009) Selenoproteins. *J.Biol.Chem.* **284** 723. PMID: 18757362.

Lu et al (2006) Inhibition of mammalian thioredoxin reductase by some flavonoids: implications for myricetin and quercetin anticancer activity. *Cancer.Res.* **66** 4410. PMID: 16618767.

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Ong & Khoo et al (1997) Biological effects of myricetin. *Gen.Pharmacol.* **29** 121. PMID: 9251891.

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Lampiran 2. Gambar alat dan bahan penelitian

Alat	
 A white analytical balance scale with a stainless steel weighing pan and a digital display showing 0.0000.	 A white and black UV-Vis spectrophotometer with a digital display and a keypad.
Neraca analitik	Spektro uv-vis
 A blue magnetic stirrer with a digital display showing 265, stirring a yellow liquid in a beaker.	 Three clear glass volumetric pipettes with red rubber bulbs, lying on a wooden surface.
Setirer	Pipet tetes

Alat



Oven



Kulkas



Viskometer



pH meter

Alat

Pipet volume



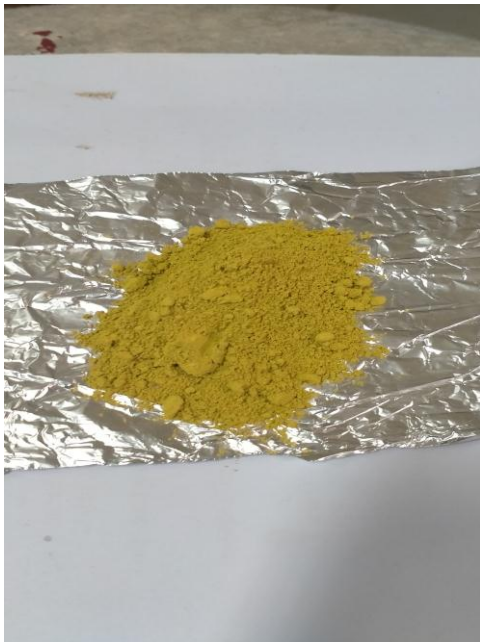
Botol serum



Batang pengaduk



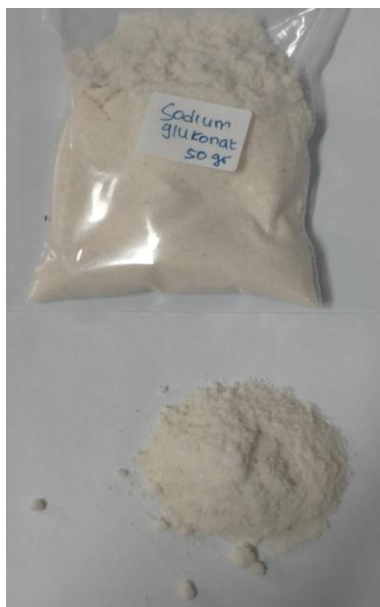
Sedok tanduk

Bahan

Serbuk myrisetin



gylcerin



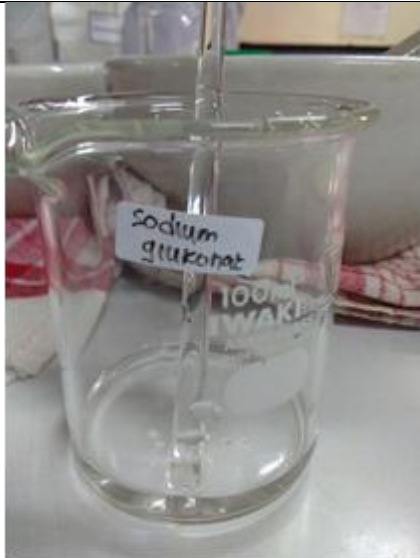
Sodium glukonat



Optipen

Bahan	
	
Xanthan gum	Aquadest
	
Hyaluronic acid	

Lampiran 3. Cara pembuatan serum myrisetin.

Cara pembuatan	
	
Gambar. Mengembangkan <i>hyaluronic acid</i>	Gambar. Mengembangkan <i>xanthan gum</i>
	
Gambar. Melarutkan <i>sodium gluconate</i>	Gambar. Gliserin



Gambar. Pencampuran semua bahan menjadi satu sampai terbentuk serum



Sediaan serum myrisetin.

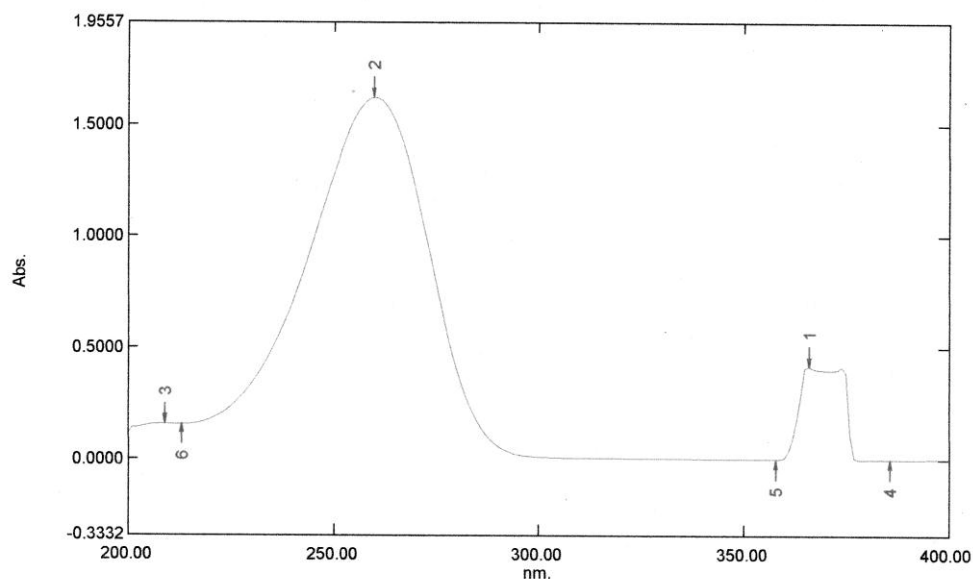
Lampiran 4. Pembuatan kurva kalibrasi dan validasi metode.

1. Penentuan panjang gelombang.

Spectrum Peak Pick Report

03/11/2020 10:37:06 AM

Data Set: File_200311_103334 - RawData



[Measurement Properties]
 Wavelength Range (nm.): 200.00 to 400.00
 Scan Speed: Medium
 Sampling Interval: 1.0
 Auto Sampling Interval: Disabled
 Scan Mode: Single

[Instrument Properties]
 Instrument Type: UV-1800 Series
 Measuring Mode: Absorbance
 Slit Width: 1.0 nm
 Light Source Change Wavelength: 340.0 nm
 S/R Exchange: Normal

[Attachment Properties]
 Attachment: None

[Operation]
 Threshold: 0.0010000
 Points: 4
 InterPolate: Disabled
 Average: Disabled

[Sample Preparation Properties]
 Weight:
 Volume:
 Dilution:
 Path Length:
 Additional Information:

No.	P/V	Wavelength	Abs.	Description
1	Ⓢ	366.00	0.4199	
2	Ⓢ	260.00	1.6198	
3	Ⓢ	209.00	0.1580	
4	Ⓢ	386.00	0.0027	
5	Ⓢ	358.00	0.0036	
6	Ⓢ	213.00	0.1562	

2. Operating Time Mirisetin.

Kinetics Data Print Report

03/11/2020 04:13:17 PM

Time (Minute)	RawData ...
0.000	0.417
1.000	0.418
2.000	0.417
3.000	0.417
4.000	0.417
5.000	0.418
6.000	0.418
7.000	0.418
8.000	0.418
9.000	0.418
10.000	0.418
11.000	0.417
12.000	0.418
13.000	0.418
14.000	0.418
15.000	0.418
16.000	0.418
17.000	0.418
18.000	0.418
19.000	0.419
20.000	0.418
21.000	0.419
22.000	0.419
23.000	0.418
24.000	0.418
25.000	0.418
26.000	0.418
27.000	0.418
28.000	0.418
29.000	0.418
30.000	0.418

3. Linieritas.

Penimbangan myrisetin :

Kertas kosong : 0,2812 g

Kertas kosong + isi : 0,2921 g

Kertas sisa : 0,2821 g

Zat aktif : 0,0100 g

Membuat larutan induk sebesar 100 ppm dengan menimbang 10,6 mg myrisetin ditambahkan etanol p.a sampai 100 mL, selanjutnya dibuat seri konsentrasi :

1) 4,4 ppm

$$C_1 \times V_1 = C_2 \times V_2$$

$$100 \text{ ppm} \times V_1 = 4,4 \text{ ppm} \times 10 \text{ mL}$$

$$V_1 = 0,44 \text{ mL}$$

2) 6,5 ppm

$$C_1 \times V_1 = C_2 \times V_2$$

$$100 \text{ ppm} \times V_1 = 6,5 \text{ ppm} \times 10 \text{ mL}$$

$$V_1 = 0,65 \text{ mL}$$

3) 8,6 ppm

$$C_1 \times V_1 = C_2 \times V_2$$

$$100 \text{ ppm} \times V_1 = 8,6 \text{ ppm} \times 10 \text{ mL}$$

$$V_1 = 0,86 \text{ mL}$$

4) 10,6 ppm

$$C_1 \times V_1 = C_2 \times V_2$$

$$100 \text{ ppm} \times V_1 = 10,6 \text{ ppm} \times 10 \text{ mL}$$

$$V_1 = 1,06 \text{ mL}$$

5) 12,4 ppm

$$C_1 \times V_1 = C_2 \times V_2$$

$$100 \text{ ppm} \times V_1 = 12,4 \text{ ppm} \times 10 \text{ mL}$$

$$V_1 = 1,24 \text{ mL}$$

Konsentrasi standar (ppm)	Absorbansi
4,4	0,355
6,5	0,465
8,6	0,580
10,6	0,682
12,4	0,793

Persamaan regresi linier antara konsentrasi (ppm) dan serapan diperoleh:

$$a = 0,1131$$

$$b = 0,0543$$

$$r = 0,9996$$

$$y = 0,1131 + 0,0543x$$

keterangan:

x = konsentrasi (ppm)

y = serapan

4. Akurasi

KONSENTRASI	REPLIKASI	ABS	KONSENTRASI	ppm sebenarnya	%	RATA-RATA	RECOVERY
80%	1	0,460	6,3836	6,5	98%	98,21%	99,76%
	2	0,461	6,4020	6,5	98%		
	3	0,459	6,3652	6,5	98%		
100%	1	0,580	8,5920	8,6	100%	99,98%	
	2	0,581	8,6104	8,6	100%		
	3	0,58	8,5920	8,6	100%		
120%	1	0,695	10,7084	10,6	101%	101,08%	
	2	0,696	10,7268	10,6	101%		
	3	0,695	10,7084	10,6	101%		

$$\% \text{ recovery} = \frac{\text{konsentrasi terukur}}{\text{konsentrasi sebenarnya}} \times 100\%$$

Hasil dari akurasi didapatkan rata-rata % recovery yaitu 98,21%, 99,98% dan 101,08%. Rata-rata % yaitu 99,76%.

5. Presisi

KONSENTRASI (PPM)	ABS	KONSENTRASI
4,4	0,382	4,9481
4,4	0,383	4,9665
4,4	0,349	4,3408
4,4	0,375	4,8193
4,4	0,352	4,3960
4,4	0,352	4,3960
4,4	0,378	4,8745
4,4	0,345	4,2672
4,4	0,368	4,6905
4,4	0,38	4,9113
RATA-RATA		0,280476
SD		4,6610
CV		0,060175

$$SD = \sqrt{\frac{\sum (x - \bar{x})^2}{n - 1}}$$

$$CV = \frac{SD}{X}$$

Hasil presisi didapatkan nilai SD sebesar 0,2804 dan nilai CVnya 0,06%.

Lampiran 5. Uji organoleptis sediaan serum.



Formula 1



Formula 2



Formulasi 3

Lampiran 6. Data pengujian pH serum myrisetin.

Formula	Replikasi	Hari ke-1	Hari ke-7	Hari ke-14	Hari ke-21
1	1	5.96	5.97	6.03	6.1
		5.96	5.98	6.06	6.12
		5.99	5.97	6.02	6.1
	Rata-rata	5.97	5.97	6.04	6.11
	SD	0.02	0.01	0.02	0.01
2	2	6.16	6.03	6.23	6.25
		6.14	6.07	6.2	6.22
		6.17	6.09	6.26	6.24
	Rata-rata	6.16	6.06	6.23	6.24
	SD	0.02	0.03	0.03	0.02
3	3	6.24	6.36	6.32	6.33
		6.27	6.31	6.28	6.33
		6.29	6.22	6.24	6.35
	Rata-rata	6.27	6.30	6.28	6.34
	SD	0.03	0.07	0.04	0.01

Lampiran 7. Data analisis *One Way ANOVA* pengujian pH.

Descriptive Statistics

	N	Mean	Std. Deviation	Minimum	Maximum
Hari	36	2.5000	1.13389	1.00	4.00
Formula	36	2.0000	.82808	1.00	3.00
pH	36	6.1628	.12821	5.96	6.36

One-Sample Kolmogorov-Smirnov Test

		Hari	Formula	pH
N		36	36	36
Normal Parameters ^{a,b}	Mean	2.5000	2.0000	6.1628
	Std. Deviation	1.13389	.82808	.12821
	Absolute	.170	.220	.145
Most Extreme Differences	Positive	.170	.220	.100
	Negative	-.170	-.220	-.145
Kolmogorov-Smirnov Z		1.022	1.318	.867
Asymp. Sig. (2-tailed)		.247	.062	.440

a. Test distribution is Normal.

b. Calculated from data.

ANOVA

pH

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	.450	2	.225	59.060	.000
Within Groups	.126	33	.004		
Total	.575	35			

Test of Homogeneity of Variances

pH

Levene Statistic	df1	df2	Sig.
1.683	2	33	.201

Lampiran 8. Data pengujian viskositas serum myrisetin.

Formula	Replikasi	Hari ke-1	Hari ke-7	Hari ke-14	Hari ke-21
1	1	1250	1150	950	650
		1200	1000	900	600
		1200	1100	900	600
	Rata-rata	1217	1083	917	617
	SD	29	76	29	29
2	2	1200	1100	900	550
		1250	1150	950	600
		1200	1150	950	600
	Rata-rata	1217	1133	933	583
	SD	29	29	29	29
3	3	1000	850	700	550
		1000	800	650	600
		1000	850	750	550
	Rata-rata	1000	833	700	567
	SD	0	29	50	29

Lampiran 9. Data analisis *One Way ANOVA* pengujian Viskositas

Descriptive Statistics

	N	Mean	Std. Deviation	Minimum	Maximum
Hari	36	2.5000	1.13389	1.00	4.00
Formula	36	2.0000	.82808	1.00	3.00
Viskositas	36	895.8333	234.63497	550.00	1250.00

One-Sample Kolmogorov-Smirnov Test

		Hari	Formula	Viskositas
N		36	36	36
Normal Parameters ^{a,b}	Mean	2.5000	2.0000	895.8333
	Std. Deviation	1.13389	.82808	234.63497
	Absolute	.170	.220	.131
Most Extreme Differences	Positive	.170	.220	.131
	Negative	-.170	-.220	-.111
Kolmogorov-Smirnov Z		1.022	1.318	.788
Asymp. Sig. (2-tailed)		.247	.062	.563

a. Test distribution is Normal.

b. Calculated from data.

Test of Homogeneity of Variances

Viskositas

Levene Statistic	df1	df2	Sig.
.843	2	33	.439

ANOVA

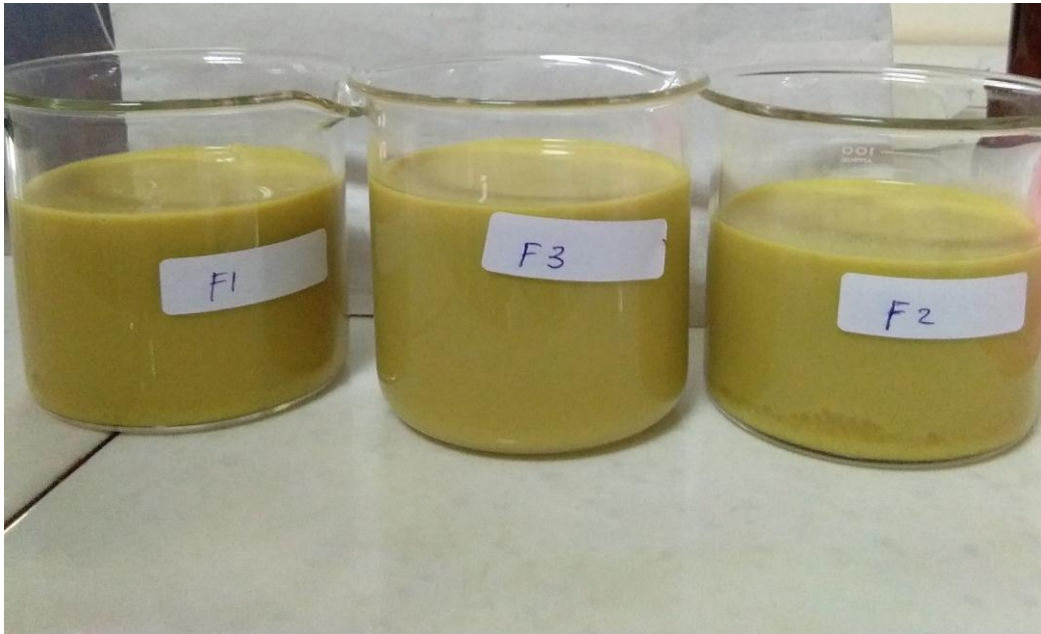
Viskositas

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	282916.667	2	141458.333	2.840	.073
Within Groups	1643958.333	33	49816.919		
Total	1926875.000	35			

Lampiran 10. Data hasil pengujian stabilitas serum myrisetin.

Formula	Siklus	Pada suhu dingin			Pada suhu panas		
		Konsistensi	Warna	Bau	Bentuk	Konsistensi	Bau
1	1	sedikit	Kuning	Khas	sedikit	Kuning	Khas
		kental	kecoklatan	myrisetin	kental	kecoklatan	myrisetin
2		sedikit	Kuning	Khas	sedikit	Kuning	Khas
		kental	kecoklatan	myrisetin	kental	kecoklatan	myrisetin
3		sedikit	Kuning	Khas	sedikit	Kuning	Khas
		kental	kecoklatan	myrisetin	kental	kecoklatan	myrisetin
1	2	sedikit	Kuning	Khas	sedikit	Kuning	Khas
		kental	kecoklatan	myrisetin	kental	kecoklatan	myrisetin
2		sedikit	Kuning	Khas	sedikit	Kuning	Khas
		kental	kecoklatan	myrisetin	kental	kecoklatan	myrisetin
3		sedikit	Kuning	Khas	sedikit	Kuning	Khas
		kental	kecoklatan	myrisetin	kental	kecoklatan	myrisetin
1	3	sedikit	Kuning	Khas	sedikit	Kuning	Khas
		kental	kecoklatan	myrisetin	kental	kecoklatan	myrisetin
2		sedikit	Kuning	Khas	sedikit	Kuning	Khas
		kental	kecoklatan	myrisetin	kental	kecoklatan	myrisetin
3		sedikit	Kuning	Khas	sedikit	Kuning	Khas
		kental	kecoklatan	myrisetin	kental	kecoklatan	myrisetin
1	4	sedikit		Khas	sedikit		Khas
		kental	Kuning gelap	myrisetin	kental	Kuning gelap	myrisetin
2		sedikit		Khas	sedikit		Khas
		kental	Kuning gelap	myrisetin	kental	Kuning gelap	myrisetin
3		sedikit		Khas	sedikit		Khas
		kental	Kuning gelap	myrisetin	kental	Kuning gelap	myrisetin
1	5	sedikit		Khas	sedikit		Khas
		kental	Kuning gelap	myrisetin	kental	Kuning gelap	myrisetin
2		sedikit		Khas	sedikit		Khas
		kental	Kuning gelap	myrisetin	kental	Kuning gelap	myrisetin
3		sedikit		Khas	sedikit		Khas
		kental	Kuning gelap	myrisetin	kental	Kuning gelap	myrisetin
1	6	sedikit		Khas	sedikit		Khas
		kental	Kuning gelap	myrisetin	kental	Kuning gelap	myrisetin
2		sedikit		Khas	sedikit		Khas
		kental	Kuning gelap	myrisetin	kental	Kuning gelap	myrisetin
3		sedikit		Khas	sedikit		Khas
		kental	Kuning gelap	myrisetin	kental	Kuning gelap	myrisetin

Lampiran 11. Hasil uji stabilitas serum myrisetin secara fisik.



Gambar sebelum uji stabilitas fisik serum myrisetin.



Gambar sesudah uji stabilitas secara fisik sediaan serum myrisetin.