BAB V
KESIMPULAN DAN SARAN

A. Kesimpulan

Dari hasil penelitian yang telah dilakukan, maka dapat disimpulkan bahwa :

Pertama, gemfibrozil yang dibuat dalam bentuk kristal sferis menggunakan metode *spherical agglomeration* (SA) menunjukkan kelarutan yang lebih baik daripada gemfibrozil murni

Kedua, didapatkan karakteristik kristal sferis gemfibrozil dengan metode *spherical agglomeration* (SA)

Ketiga, formula sediaan kristal gemfibrozil yang memiliki bentuk sferis dengan sempurna yaitu F1(gemfibrozil + PVP 2%)

B. Saran

Pertama, perlu dilakukan pengujian kristalinitas seperti DSC pada gemfibrozil sferis

Kedua, perlu dilakukan pengujian sifat alir untuk mengetahui perbandingan sifat alir dari gemfibrozil murni dan gemfibrozil sferis

Ketiga, perlu dilakukan dengan metode lain seperti metode *perubahan solvent*(SC), Metode *Difusi Kuasi Emulsi* (QESD), Metode *Netralisasi* (NM),dan Metode *Co-Agglomerasi* (CCA), Metode *Difusi Amonia* (AD)

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LAMPIRAN

Lampiran 1 COA

Phapros

022/S.Pr/PPPP-LPP/V/19
Semarang, 11 Mei 2019

Kepada Yth:
Fakultas Farmasi
Universitas Setia Budi
d/a Jl. Let. Jend. Sutoyo – Solo 57127
Telp. 0271 - 852518
Up. Ibu Prof. Dr. R.A Oetari,SU., MM., M.Sc., Apt

Perihal : Permohonan Bahan Baku

Dengan hormat,
Memenuhi permintaan Ibu sesuai surat no. 4524/A10-4/05.04.2019 per tgl. 5 April 2019 perihal tersebut di atas, bersama ini kami kirimkan :

No.	Nama bahan baku	Um	Jumlah	Certificate Of Analisys
1	Gemfibrozil	Gr	100	√
2				

Untuk keperluan penelitian Mahasiswa :

No.	Nama	NIM
1	Retna Rosiana Dewi	21154479A

Mohon diterima dengan baik dan selanjutnya apabila penelitian telah selesai, agar mengirimkan 1 eksemplar laporan untuk keperluan perpustakaan kami.

Demikian, semoga bermanfaat dan terima kasih.

Hormat Kami,

Phapros

Drs. Giri Hardiyatmo, Apt, MM
Manager PPIC

Diterima oleh :
Tanggal :
Tanda tangan :

OFFICE :
PT. Phapros Tbk
Gedung RNI
Jl. Denpasar Raya Kav DIII
Kuningan, Jakarta 12950. INDONESIA
Phone: (62-21) 527 6262, 252 3820
Fax: (62-21) 526 6361
E-mail:marketing@phapros.co.id
Website:http://www.phapros.co.id

FACTORY :
PT. Phapros Tbk
Jl. Simongan 131
Semarang 50148. INDONESIA
Phone: (62-24) 7603021 (hunting)
Fax: (62 24) 760 5153, (62 24) 760 6872
P.O. Box: 1233
E-mail:factory@phapros.co.id
Website:http://www.phapros.co.id

xxpfrpx.p
Page: 1

37.0.8 Test result Report (wdf)*
PHAPROS, PT

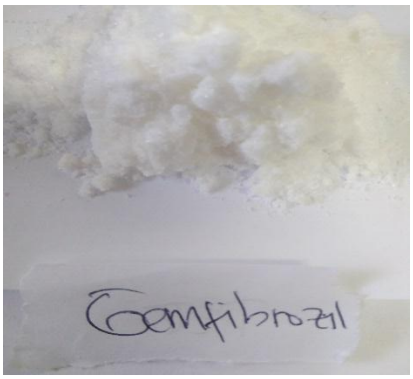
Date: 13/05/18
Time: 12:32:53

Quality Order	Batch	Item Number	Insp Loc	Location	Procedure	Qty Pending	Qty Accepted	Qty Rejected	Order Date	Due Date	Eff Date	S
BB.18/0871	1E227	1430715S GEMFIBROZIL	PK	GSB	Pemeriksaan BB/BK	250.0	250.0	0.0	06/03/18	16/03/15	14/03/18	C

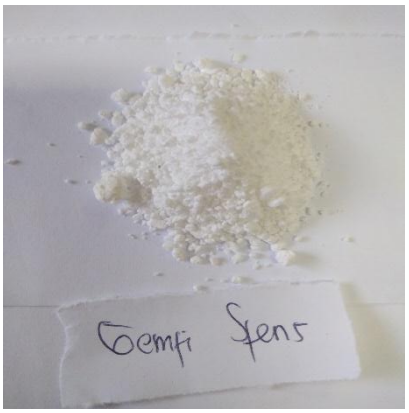
Qc Number	Characteristic	Actual Results	Specification	Measure	Pass
200 01	PEMERIAN	SESUAI	*		yes
02	KELARUTAN	SESUAI	*		yes
03	IDENTIFIKASI	SESUAI	*		yes
04	JARAK LEBUR 56.6 - 58.6	58.6	58;61	°C	yes
05	LOGAM BERAT <20	20	<=20	µg/g	yes
06	KEMURNIAN KROMATOGRAFI	SESUAI	SESUAI		yes
07	KADAR AIR	0.139	<=0.25	PERSEN	yes
08	KADAR	100.979	98;102	PERSEN	yes
091	SISA RESIDU METHANOL COA -	-	<=1000	µg/g	yes
092	SISA RESIDU THF NOT DETECTED	-	<=720	µg/g	yes
093	SISA RESIDU TOLUENE -	-	<=890	µg/g	yes
094	SISA RESIDU ACETONE -	-	<=1000	µg/g	yes
101	PETUGAS SAMPLING	CEC,HAK	*		yes
102	PEMERIKSA	HAA,CEC,SAU	*		yes
11	CATATAN	-	*		yes

Lampiran 2 Gambar serbuk

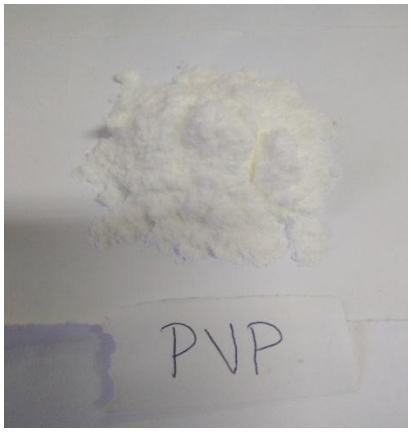
- Gemfibrozil serbuk



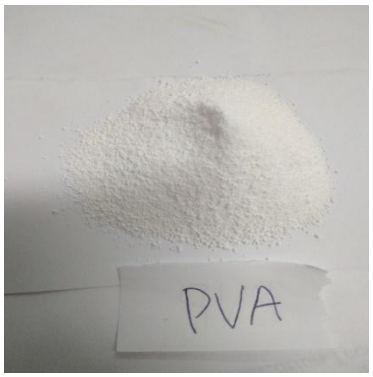
- Gemfibrozil sferis



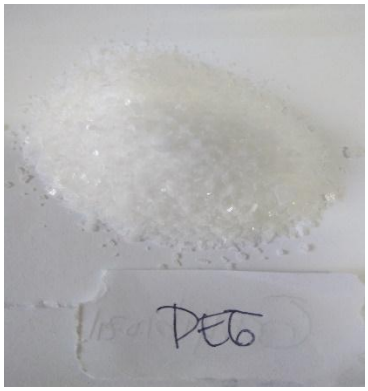
- PVP



- PVA



- PEG 4000



Lampiran 3 Kurva kalibrasi

- Larutan Induk

- Larutan stock

- Seri konsentrasi

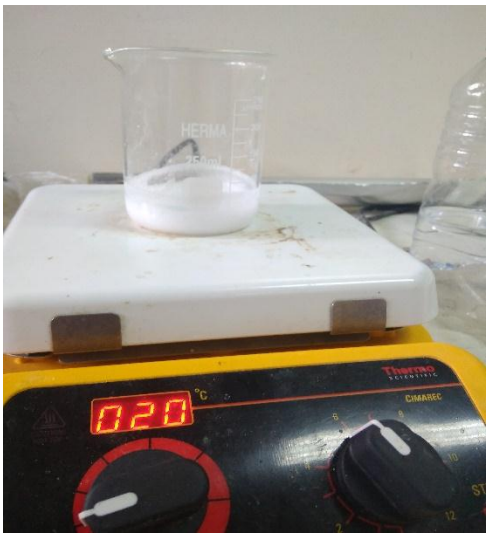
Lampiran 4 Alat Penelitian

- Stirrer

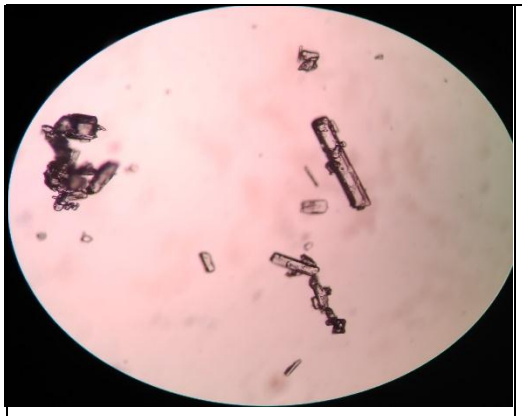
- Mikroskop optik

- Spektro UV-Vis

Lampiran 5 Foto Larutan Sferis



Lampiran 6 Foto Mikroskop Optik

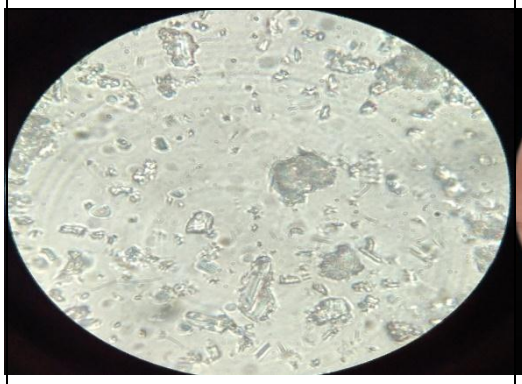


A

Gemfi murni

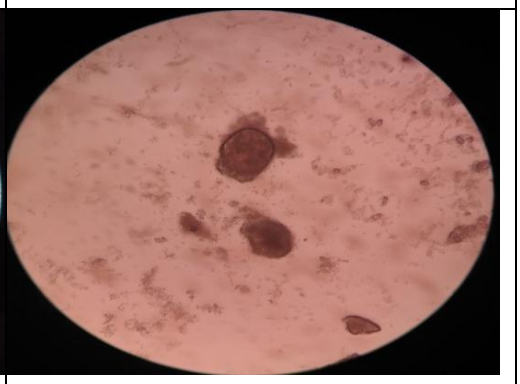
B

Gemfi + PVP 2%



C

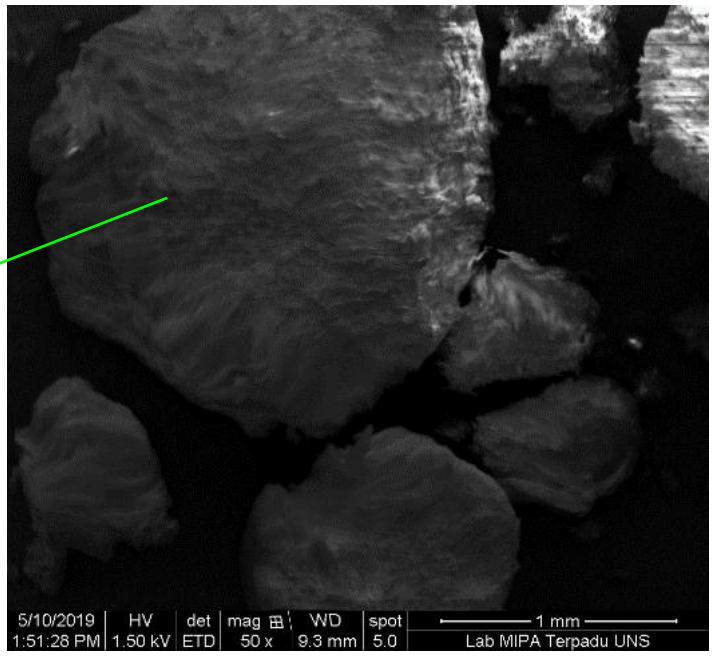
Gemfi + PVA 2%



D

Gemfi + PEG 4000 2%

Lampiran 7 Foto Hasil SEM



Lampiran 8 Perhitungan Penentuan % Rendemen

- Pernimbangan gemfibrozil murni

Kertas kosong = 0,2789

Kertas + zat = 2,2805

Kertas sisa = 0,2798 -

2,0007 gram

- Penimbangan gemfi sferis

Kertas saring kosong = 0,2815

Kertas saring + zat = 2,1459

Kertas saring sisa = 0,2828 -

1,8683 gram

$$\% \text{ Rendeman} = \frac{bobot\ kristal\ sferis}{bobot\ serbuk} \times 100\%$$

$$= \frac{1,8631}{2,0007} \times 100\%$$

= 93,117 %

Lampiran 9 Perhitungan Kelarutan

- Gemfibrozil Murni

  Penimbangan serbuk gemfibrozil murni :

  Kertas kosong = 0,2674 gram

  Kertas + zat = 0,3234 gram

  Kertas sisa = 0,2734 gram -
  0,050 gram

- Gemfi sferis

  Penimbangan serbuk sferis gemfibrozil :

  Kertas kosong = 0,2693 gram

  Kertas + zat = 0,3243 gram

  Kertas sisa = 0,2743 gram -
  0,050 gram

No	Obat	Kadar (ppm)	Kadar (mg)
1.	Gemfibrozil Murni	55,704	2,285
2.	Krital sferis Gemfibrozil	146,143	7,307

Perhitungan kadar :

1. Gemfibrozil murni =

   y = a + bx

   0,329 = 0,0068667 + 0,0057829 x

   $x = \frac{0,329 - 0,0068667}{0,0057829} = 55,704\ ppm$

   kadar (mg) = $\frac{55,704\ mg}{1000\ mL} \times 5 \times 50\ mL = 2,785\ mg$

2. Gemfibrozil sferis =

   y = a + bx

   0,852 = 0,0068667 + 0,0057829 x

   $x = \frac{0,852 - 0,0068667}{0,0057829} = 146,143\ ppm$

   kadar (mg) = $\frac{146,143\ mg}{1000\ mL} \times 5 \times 50\ mL = 7,307\ mg$

Lampiran 10 Perhitungan kurva kalibrasi larutan standar gemfibrozil

- Larutan Induk 1000 ppm ⟶ 10 mg/10 mL

Penimbangan :

Kertas kosong = 0,2662

Kertas + zat = 0,2765

Kertas sisa = 0,2665 -

0,0100 gram

- Pembuatan larutan lamda maks

100 ppm dari induk 1000 ppm

V1 x C1 =V2 x C2

V1 x 1000 ppm = 10 ml x 100 ppm

V1 = 1 mL

Lampiran 11 Perhitungan Seri konsentrasi

Konsentrasi	Perhitungan
20 ppm	V1 x C1 =V2 x C2 V1 x 1000 ppm = 10 ml x 20 ppm V1 = 0,2 mL
40 ppm	V1 x C1 =V2 x C2 V1 x 1000 ppm = 10 ml x 40 ppm V1 = 0,4 mL
60 ppm	V1 x C1 =V2 x C2 V1 x 1000 ppm = 10 ml x 60 ppm V1 = 0,6 mL
80 ppm	V1 x C1 =V2 x C2 V1 x 1000 ppm = 10 ml x 80 ppm V1 = 0,8 mL
100 ppm	V1 x C1 =V2 x C2 V1 x 1000 ppm = 10 ml x 100 ppm V1 = 1 mL
120 ppm	V1 x C1 =V2 x C2 V1 x 1000 ppm = 10 ml x 120 ppm V1 = 1,2 mL

Lampiran 12 Verifikasi Metode Analisis

- **Linearitas**

Data serapan gemfibrozil dalam berbagai konsentrasi pada medium dapar phospat 7,5 pada panjang gelombang maksimal : 274 nm

Konsentrasi (ppm)	Absorbansi	Persamaan
20	0,123	a = 0,0068667 b = 0,0057829 r = 0,999602
40	0,244	
60	0,352	
80	0,462	
100	0,58	
120	0,709	

- **Penentuan LOD & LOQ**

X (ppm)	Y (abs)	Y'	|Y-Y'|	|Y-Y'|2
20	0,123	0,1225238	0,0005	0,00000022675736961
40	0,244	0,238181	0,0058	0,00003386131519274
60	0,352	0,3538381	-0,0018	0,00000337859410431
80	0,462	0,4694952	-0,0075	0,00005617859410431
100	0,58	0,5851524	-0,0052	0,00002654702947846
120	0,709	0,7008095	0,0082	0,00006708390022676
			∑|Y-Y'|2	0,0002

Nilai Y' diperoleh dari subtitusi konsetrasi dalam persamaan y = 0,0068667-0,0057829x, dengan nilai x adalah konsentrasi (ppm) dan y adlahan serapan (Y')

$$S_{x/y} = \sqrt{\frac{\sum|Y-Y'|2}{N-2}}$$

Keterangan :

$S_{x/y}$ = Simpangan baku residual

N = Jumlah data

∑|Y-Y'|2 = Jumlah kuadrat total residual

$$S_{x/y} = \sqrt{\frac{0,0002}{6-2}} = 0,007071$$

LOD = $\frac{3,3\ (0,007071)}{0,0057829}$

= $\frac{0,0233343}{0,0057829}$ = 4,0350 ppm

Serapan LOD

y = 0,0068 + 0,007829 (4,0350)

= 0,0068 + 0,03159

= 0,03839

LOQ = $\frac{10(0,007071)}{0,0057829}$

= $\frac{0,07071}{0,0057829}$ = 12,2274 ppm

Serapan LOQ

y = 0,0068 + 0,007829 (12,2274)

= 0,0068 + 0,09572

= 0,1025

- **Presisi**

Konsentrasi	ABS	Konsentrasi(ppm)
100	0,583	99,627
100	0,578	98,762
100	0,587	100,319
100	0,585	99,973
100	0,583	99,627
100	0,598	102,221
100	0,588	100,492
100	0,584	99,800
100	0,586	100,146
100	0,593	101,356
Rata-rata		100,232
SD		0,996

- Perhitungan CV = $\frac{SD}{rata-rata} \times 100\%$

= $\frac{0,996}{100,232} \times 100\% =$ 0,993 %

- **Akurasi**

Konsentrasi (ppm)	Absorbansi
20	0,123
40	0,244
60	0,352
80	0,462
100	0,580
120	0,709

Konsentrasi (ppm)	Replikasi	absorbansi	konsentrasi	Recorvery	Rata-rata
80	1	0,456	77,6663	97%	95,28 %
	2	0,438	74,5537	93%	
	3	0,449	76,4559	96%	
100	1	0,580	99,1090	99%	94,40 %
	2	0,583	99,6278	100%	
	3	0,582	99,4549	99%	
120	1	0,759	130,0626	108%	107,91%
	2	0,752	128,8521	107%	
	3	0,756	129,5438	108%	
Rata-rata					99,196%

Lampiran 13 Panjang Gelombang Maksimum

Spectrum Peak Pick Report

05/17/2019 02:37:49 PM

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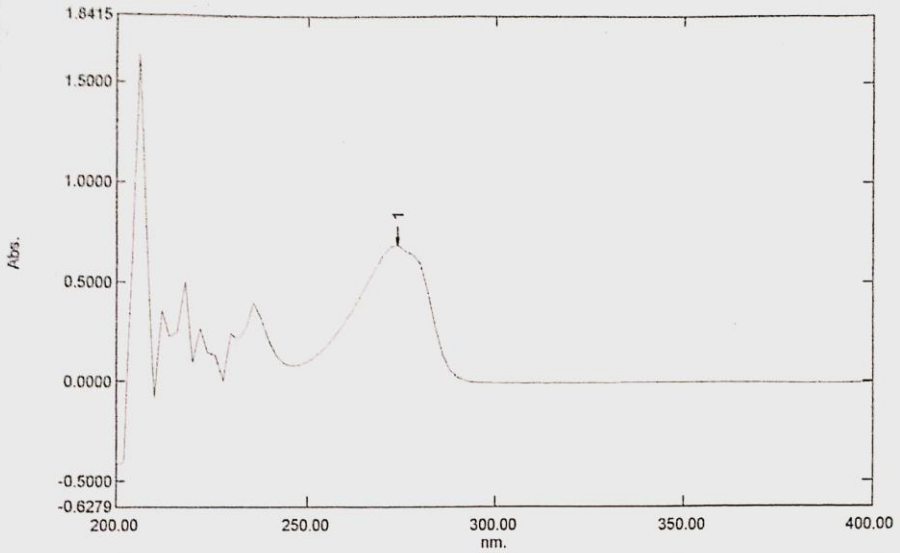

No.	P/V	Wavelength	Abs.	Description
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2		308.00	-0.0117	
3		246.00	0.0758	

[Measurement Properties]
Wavelength Range (nm.): 200.00 to 400.00
Scan Speed: Fast
Sampling Interval: 2.0
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:

Page 1 / 1

Lampiran 14 Opperating time

Kinetics Data Print Report

05/17/2019 04:13:30 PM

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9.000	0.742
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7.000	0.741
6.000	0.742
5.000	0.741
4.000	0.741
3.000	0.741
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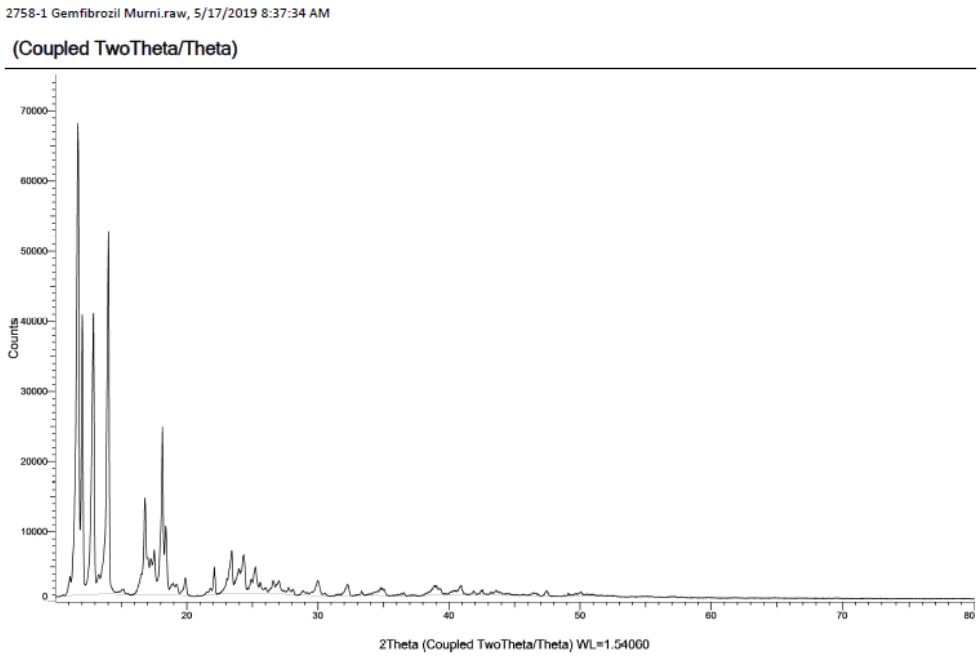
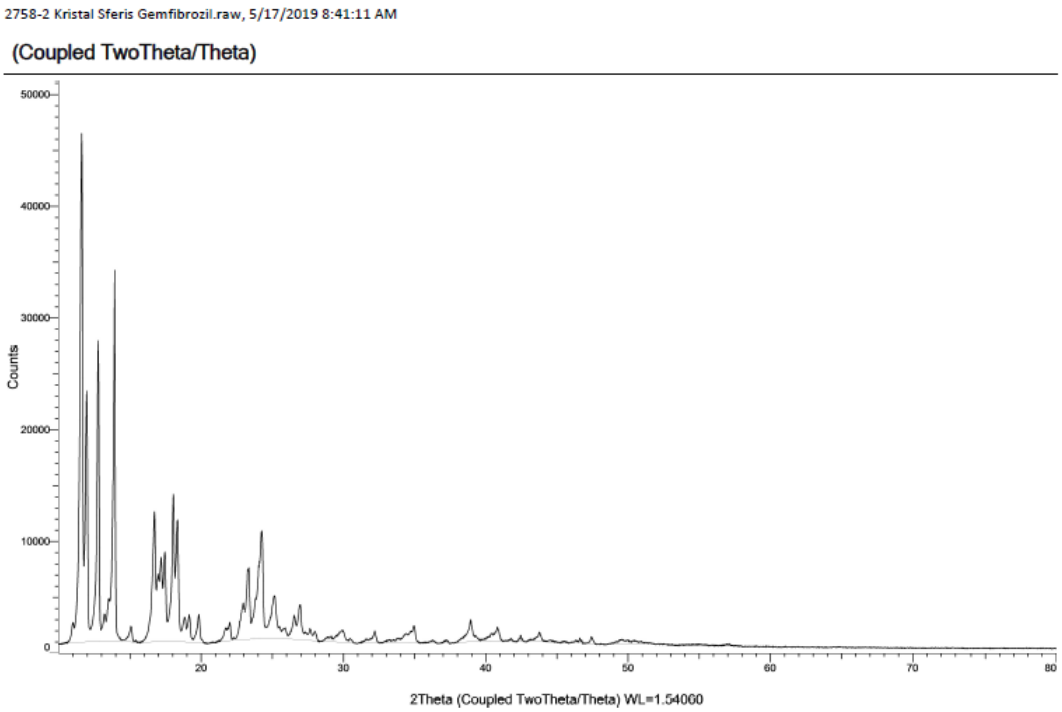
0.000	0.740

Lampiran 15 Hasil XRD

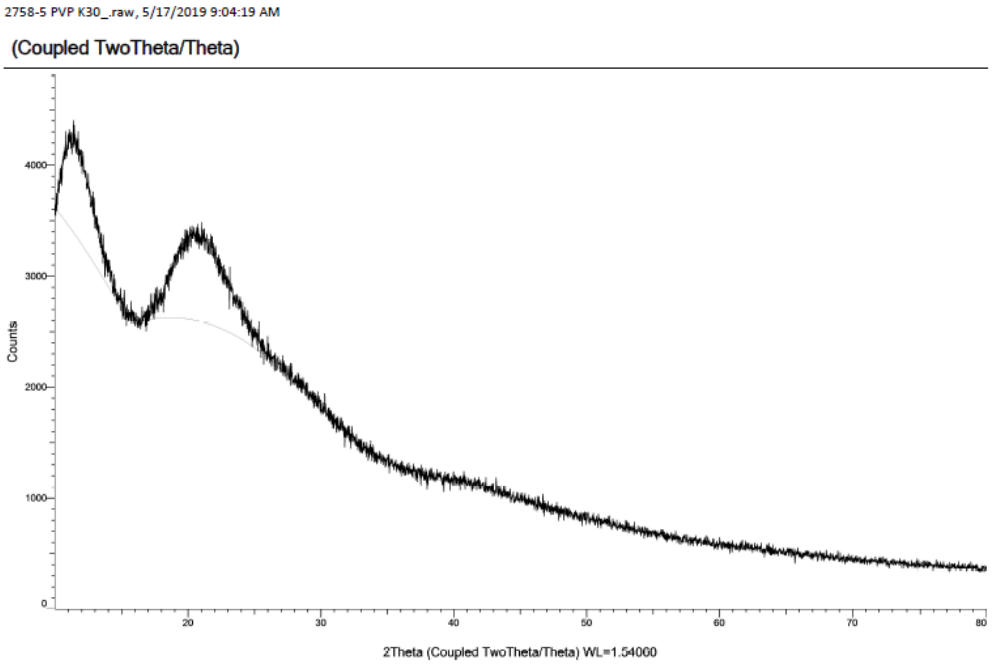
1. Kristal gemfibrozil murni



2. Kristal gemfibrozil sfersi



3. PVP K-30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(Coupled TwoTheta/Theta)
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3000
2000
1000
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20
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50
60
70
80
2Theta (Coupled TwoTheta/Theta) WL=1.54060