

## CHAPTER III

### PRELIMINARY STUDY ON PHYSICAL PROPERTIES OF COMPOSITE PARTICLES OF RICE FLOUR OR RICE STARCH WITH MICROCRYSTALLINE CELLULOSE

#### Introduction

The objectives in the development of directly compressible diluents (DC diluents) are usually to improve at least one of these properties such as compressibility, flowability, and disintegration properties. Until now, the development of DC diluents is not searching for a new one, but usually modification of the existing excipient(s) to get the new one with an improvement of its properties and/or the combined properties of each excipient.

In this study, two cellulose types, powder cellulose (Vitacel<sup>®</sup>) and microcrystalline cellulose (MCC) from two sources (Avicel<sup>®</sup> and Vivapur<sup>®</sup>) were compared for their physical properties and selected for the production of coprocessed excipient between native starch and cellulose using spray drying technique. The cellulose type with suitable physical properties and reasonable price was chosen to combine with native starch. Native starch source in this experiment came from rice and was divided into two types, namely rice flour (RF) and rice starch (RS). Because the ratio of the combining excipients and particle size also influence the physical properties of powders and tablets, the effect of starch and MCC ratio and the particle size of MCC used were therefore investigated. The objectives of the study described in this chapter are:

1. To produce the composite particles of starch and MCC via spray drying technique
2. To determine the effect of starch types used, i. e. rice flour and rice starch, and ratio of starch and MCC on the physical and tableting properties of the obtained powders and tablets
3. To determine the effect of particle size of MCC used on the physical and tableting properties of the obtained powders and tablets
4. To compare the powder characteristics and compressibility of the coprocessed excipients with other DC diluents

## Materials and Methods

### Materials

Rice flour	(Cho Heng Co., Ltd., Thailand)
Rice starch	(Cho Heng Co., Ltd., Thailand)
Microcrystalline cellulose	(Vivapur <sup>®</sup> 101, Lot No. 5610102917, J.Rettenmaier & Söhne, Germany; Avicel <sup>®</sup> 101, Lot No. 1824, Asahi Chemical Industry Co., Ltd., Japan)
Powder cellulose	(Vitacel <sup>®</sup> , Control No. 0781770415, J. Rettenmaier & Söhne, Germany)
Eratab <sup>®</sup>	(Lot No. T440219, Erawan Pharmaceutical Research & Laboratory Co., Ltd., Thailand)
Tabletose <sup>®</sup>	(Lot No. L0021A4003, Meggle GMBH, Germany)
Cellactose <sup>®</sup>	(Lot No. L0016A4901, Meggle GMBH, Germany)
Deionized water	

### Methods

#### 1. Tableting properties of cellulose

Powder cellulose (Vitacel<sup>®</sup>) or two MCC (Avicel<sup>®</sup> and Vivapur<sup>®</sup>) powder of  $500 \pm 5$  mg was accurately weighed and compressed on punch-die assembly using a hydraulic press at compression force of 500 lb. Flat-faced punch and die (12.7 mm in diameter) were used for tableting. The resultant tablets were evaluated hardness, thickness, diameter, percent friability, and disintegration time as in 5.1 to 5.3.

#### 2. Preparation of Spray Dried Formulations

All starches were dried in hot air oven (Type ULSO, Memmert, Germany) at 80° C for two hours before use. Microcrystalline cellulose (MCC) from Vivapur<sup>®</sup> was divided into two categories, VS and VJM. VS was prepared by sieving MCC through a sieve with mesh number of 325 which had an opening aperture of 45  $\mu$ m (Endecotts Ltd., England). VJM was MCC of which particle size was reduced by jet milling (Current Jet Crusher, CJ-10, Isekyu Co., Ltd., Japan).

## 2.1 Rice Flour and MCC Formulations

Rice flour (RF) and VS or VJM were weighed according to the compositions shown in Table 3-1. Deionized water (DIW) was weighed and added to make the final solid content of 20% w/w. Then the suspension was mixed thoroughly with the aid of homogenizer (Ultra - turrax T50 DPX, IKA, Germany) for 10 minutes to obtain homogeneous suspension. The suspension was subsequently spray dried by spray dryer (Niro Atomizer, Denmark). The spray drying conditions used were as follows: inlet air temperature of 130°C, atomizing air pressure of 2 bars and feed rate of 25-28 g/min. Spray-dried powders were evaluated physical and tableting properties as in 4 and 5.

## 2.2 Rice Starch and MCC Formulations

Rice starch (RS) was employed to replace rice flour to prepare composite particles (Table 3-1). The quantities of materials, process of the preparations and condition used in spray drying were the same as those for rice flour formulations as described in 1.1. Spray-dried powders were evaluated physical and tableting properties as in 4 and 5.

## 3. Tableting properties of selected composite particles and commercial DC diluents

Selected composite particles and commercially available DC diluents were compressed and compared their tableting properties. Physical properties of powders were evaluated as in 4. Tablets preparation and evaluation were the same as in 5.

## 4. Physical Properties of Powders

### 4.1 Loss on Drying (LOD)

Powder samples of 1.5 g were accurately weighed on the pan of moisture analyzer (Model MA30, Sartorius, Germany). The temperature and conditions were set at 105°C, Auto and 0-100% Mode. The sample was dried to constant weight, and percent LOD was read. The mean and standard deviation of three determinations were calculated.

### 4.2 Shape, Size, and Surface Topography

The powders were sputter-coated with gold prior to the microscopic

Table 3-1 The compositions of spray dried formulations.

Formulations		Ratio Starch : MCC	RF or RS (g)	VS or VJM (g)	DIW <sup>3</sup> (g)
RF <sup>1</sup>	RS <sup>2</sup>				
RFSD <sup>4</sup>	RSSD <sup>4</sup>	—	400	—	2000
RFVS-91 <sup>5</sup>	RSVS-91	9 : 1	360	40	2000
RFVJM-91	RSVJM-91				
RFVS-82	RSVS-82	8 : 2	320	80	2000
RFVJM-82	RSVJM-82				
RFVS-73	RSVS-73	7 : 3	280	120	2000
RFVJM-73	RSVJM-73				
RFVS-64	RSVS-64	6 : 4	240	160	2000
RFVJM-64	RSVJM-64				
RFVS-55	RSVS-55	5 : 5	200	200	2000
RFVJM-55	RSVJM-55				

- Note : 1 = rice flour.  
 2 = rice starch.  
 3 = deionized water.  
 4 = SD at the end of RF and RS denotes that material is processed by spray drying.  
 5 = The number behind hyphen indicates the proportion of each combined material : the first number is for RF or RS and the second one is for VS or VJM, for example, RFVS-91 is RF : VS in the ratio of 9 : 1.

Table 3-2 Degree of flowability and flowability index.

Degree of Flowability	Flowability Index	Angle of Repose		Compressibility		Angle of Spatula		Cohesion	
		Degree	Index	%	Index	Degree	Index	%	Index
Very Good	90-100	≤ 25	25	≤ 5	25	≤ 25	25		
		26-29	24	6-9	23	26-30	24		
		30	22.5	10	22.5	31	22.5		
Fairly Good	80-89	31	22	11	22	32	22		
		32-34	21	12-14	21	33-37	21		
		35	20	15	20	38	20		
Good	70-79	36	19.5	16	19.5	39	19.5		
		37-39	18	17-19	18	40-44	18		
		40	17.5	20	17.5	45	17.5		
Normal	60-69	41	17	21	17	46	17		
		42-44	16	22-24	16	47-59	16		
		45	15	25	15	60	15	≤ 6	15
Not Good	40-59	46	14.5	26	14.5	61	14.5	6-9	14.5
		47-54	12	27-30	12	62-74	12	10-29	12
		55	10	31	10	75	10	30	10
Bad	20-39	56	9.5	32	9.5	76	9.5	31	9.5
		57-64	7	33-36	7	77-89	7	32-54	7
		65	5	37	5	90	5	55	5
Very Bad	0-19	66	4.5	38	4.5	91	4.5	56	4.5
		67-89	2	39-45	2	92-99	2	57-79	2
		90	0	≥ 45	0	≥ 99	0	≥ 79	0

Source : Operating instructions manual of powder characteristics tester, Hosokawa Micron Corp.

examination. Shape, size, and surface topography of the powders were observed using scanning electron microscope (SEM, JSM-5410LV, Jeol, Ltd., Japan) and photomicrographs were taken at appropriate magnifications.

#### 4.3 Powder Characterization

Angle of repose, angle of spatula, bulk density, packed density, % compressibility and % cohesion of the powders were determined by powder characteristics tester (Model PT-N, Hosokawa Micron Corporation, Japan). Each value determination was assigned an index numbers determined by its measured value. The summation of these index number is flowability index which describes degree of flowability. The higher value of the flowability index indicates the better flowability of the material as shown in Table 3–2.

#### 4.4 Particle Size Measurement

Particle size and size distribution of raw materials and spray dried powders were determined in three reading by light scattering method (laser particle size distribution analyzer, Model Mastersizer-S, Malvern Instruments Ltd., UK). Half of the dispensing spoon of the sample was dispersed in approximate 200 ml of absolute ethanol which was used as dispersion medium.

### 5. Tableting Properties

Powder samples of  $500 \pm 5$  mg of each sample were accurately weighed and compressed on punch-die assembly using a hydraulic press at compression force of 2000 lb. Flat-faced punch and die (12.7 mm in diameter) were used for tableting. The obtained tablets were subjected to evaluation as follows:

#### 5.1 Hardness, Thickness and Diameter

Hardness, thickness, and diameter were determined by tablet hardness tester (Model TBH30, Erweka, Germany). Mean and standard deviation were calculated from ten determinations.

## 5.2 Percent Friability

Percent friability of twenty tablets was measured using Roche Friabilator (Erweka, Germany).

## 5.3 Disintegration Time

The disintegration time of tablets was determined in deionized water using USP disintegration test apparatus (Model ZT31, Erweka, Germany). The deionized water was kept at  $37^{\circ} \pm 1^{\circ}\text{C}$  during the test, and the disintegration test was performed without disc. The mean and standard deviation of six determinations for each formulations were calculated.

# 6. Physicochemical Characterization of Powders

## 6.1 Differential Scanning Calorimetry (DSC)

DSC thermograms were determined by differential scanning calorimeter (Model DSC7, Perkin – Elmer, USA). The small amount of powder suspension (approximately 20% w/w) containing 3 to 3.2 mg of total solid was transferred to the DSC pan which was hermetically sealed. Scans were performed at the temperature range of  $50^{\circ}$  -  $150^{\circ}\text{C}$  with the scanning rate of  $10^{\circ}\text{C}/\text{minute}$ .

Calibration of the instrument was carried out with high-purity metal with accurately known enthalpy of fusion and melting point. The calibrant used is Indium ( $\Delta H_{\text{fusion}} = 28.45 \text{ J/g}$ , melting point =  $156.6^{\circ}\text{C}$ ).

## 6.2 X-ray Powder Diffractometry

X-ray diffractograms were obtained using X-ray powder diffractometer (Model JDX – 8030, JEOL, Japan) with Ni – filtered  $\text{CuK}_{\alpha}$  radiation, a voltage of 45 kV and a current of 30 mA. X – ray diffractograms were scanned with the  $2\theta$  angle of  $5^{\circ}$  to  $60^{\circ}$ , with a step angle of  $0.04^{\circ}$ .

## 6.3 FT – Infrared Spectroscopy

The infrared spectra were determined by FTIR spectrometer (Model 1760X, Perkin Elmer, USA) using potassium bromide disk technique.

## Results and Discussion

The physical and chemical properties of RF and RS are shown in Table 3-3. The difference is the amount of protein in the starch, which is higher in RF (around 6.5 to 7.0%) than in RS (0.70%). The particle size and shape of RF and RS are also illustrated in Figure 3-1. SEM photomicrographs reveal that starch grain is polygonal in shape, and the particle size of the grain is very small (lower than 10  $\mu\text{m}$ ). RF is composed of various sizes of aggregated starch grains with larger particle size than RS. RS grains form less aggregation and are separated individually. Although the particle sizes of RS is smaller than that of RF as shown in Table 3-4, the particle sizes measured by laser diffraction are not represented the particle sizes obtained from SEM photomicrographs. This is due to the real distribution of RF and RS are bimodal (see appendix 28). The aggregation of starch grains in RF can explained by the protein content in starch that promotes physical interaction of the grains to form starch aggregates. This result agrees with a previous study by Mitchell (1994) who investigated the behavior of rice starch by drying rice starch with various amount of native protein. When rice starch with higher protein content was used (at 6.0 %), the aggregate formation of starch grains were observed. At low protein content ( $< 0.5\%$ ), RS tended to separate as individual starch grains. This protein would coat on the surface of starch and decrease the brightness of starch powder. Furthermore, protein also affects the gelatinization temperature and starch viscosity due to reduction of solvation by water molecules. Therefore, starch granules can not swell to their full capacity resulting in the raising of gelatinization temperature and the lowering of starch viscosity (Weechanrangan, 1995).

In order to select cellulose type to be used for the preparation of composite particles with rice flour or rice starch, tablets properties from two MCC brands (Avicel<sup>®</sup> 101 and Vivapur<sup>®</sup>101) and powder cellulose (Vitacel<sup>®</sup>) were investigated and tabulated in Table 3-5. Hardness and % friability of Avicel<sup>®</sup> and Vivapur<sup>®</sup> were similar while Vitacel<sup>®</sup> gave lower hardness value and % friability higher than 1%. This was due to the difference in the manufacturing process between MCC and powder cellulose as tabulated in Table 3-6. Avicel<sup>®</sup> and Vivapur<sup>®</sup> are prepared by acid hydrolysis of cellulose pulp while Vitacel<sup>®</sup> is only grinded and sieved cellulose pulp without any treatment. Acid

Table 3-3 Physical and chemical properties of RF and RS.

Topics	TP* (1993)	JPXXIII** (1996)	PhEur***	RF <sup>1</sup>	RS <sup>1</sup>
Loss on Drying (%w/w)	≤ 15.0 %	≤ 15.0 %	≤ 15.0 %	12.00 – 13.00%	12.00 – 13.00%
Amylose	--†	--†	--†	--‡	26.00%
Fat	--†	--†	--†	0.30%	0.20%
Sulfated Ash (%w/w)	≤ 1.0 %	≤ 1.0 %	≤ 1.0 %	0.30%	0.50%
Protein	--†	--†	--†	6.50 – 7.00 %	0.70%
Carbohydrate	--†	--†	--†	79.65 – 80.65%	85.50%
pH	--†	--†	--†	6.0 – 7.0	6.0 – 7.0
Sulfur Dioxide	--†	--†	--†	None	None
Particle Size	--†	--†	--†	Not over 160µm	--‡
Maximum Gelatinization Temperature (°C)	--†	--†	--†	93°C	89°C
Color of Powder (Lightness)	--†	--†	--†	93.20	99.58

Note : \* = data from Thai Pharmacopoeia, 1993

\*\* = data from Japanese Pharmacopoeia, 1996

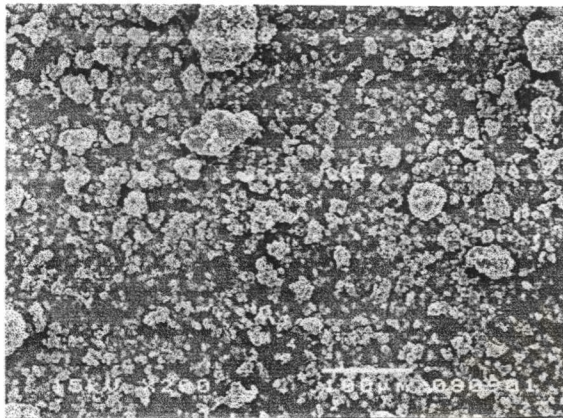
\*\*\* = data from Handbook of Pharmaceutical Excipients (Kippe , 2000)  
and European Pharmacopoeia (1984)

† = not specified

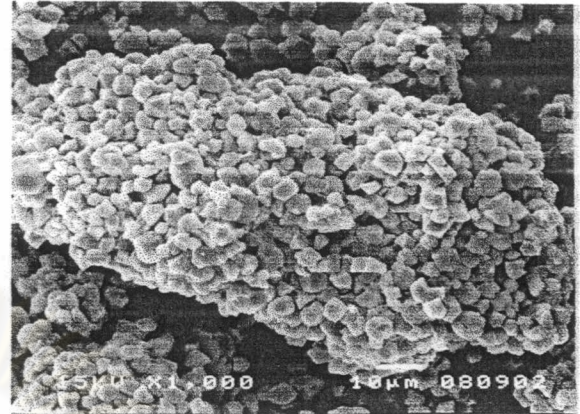
‡ = not determined

1 = data from certificate of analysis of rice flour and rice starch

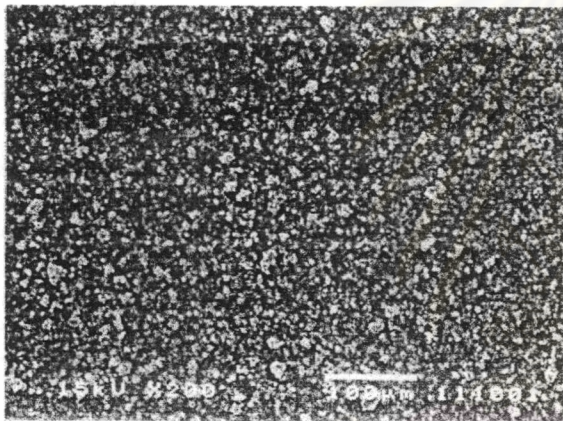




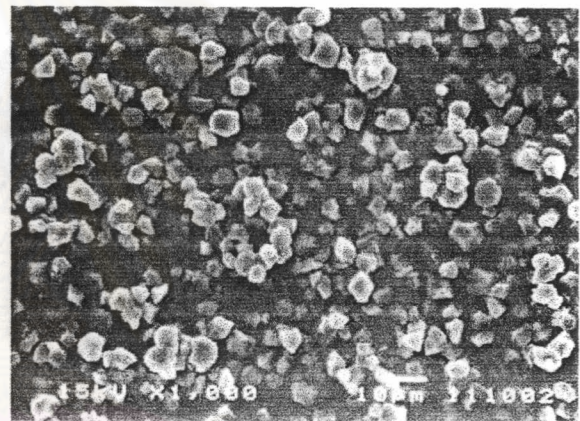
RF X 200



RF X 1000



RS X 200



RS X 1000

Figure 3-1 SEM photomicrographs of rice flour (RF) and rice starch (RS).

Table 3-4 Particle size distribution of RF and RS.

Materials	D(v,0.1)	D(v,0.5)	D(v,0.9)	Span = (D90-D10)/D50
	( $\mu\text{m}$ ) average (SD)	( $\mu\text{m}$ ) average (SD)	( $\mu\text{m}$ ) average (SD)	
RF	5.03 (0.38)	46.22 (0.56)	137.66 (0.96)	2.87 (0.06)
RS	0.56 (0.01)	13.57 (0.11)	77.51 (2.16)	5.67 (0.11)

D(v, 0.1) = 10% of the distribution is below this value

D(v, 0.5) = 50% of the distribution is below this value

D(v, 0.9) = 90% of the distribution is below this value

Table 3-5 Physical properties of MCC and powder cellulose tablets prepared by hydraulic press at compression force of 500 lb.

MCC or Powder Cellulose	Hardness (N) average (SD)	Diameter (mm) average (SD)	Thickness (mm) average (SD)	Friability (%)	DT* (min) average (SD)
Avicel® 101	104.48 (1.67)	12.81 (0.02)	4.51 (0.03)	0.40	3.40 (1.21)
Vivapur® 101	101.63 (2.45)	12.81 (0.01)	4.37 (0.05)	0.50	12.53 (1.87)
Vitacel®	71.51 (4.32)	12.77 (0.01)	4.58 (0.06)	1.17	> 30

Note \* = Disintegration time

Table 3-6 Process steps and price of Avicel®101, Vivapur®101, and Vitacel®.

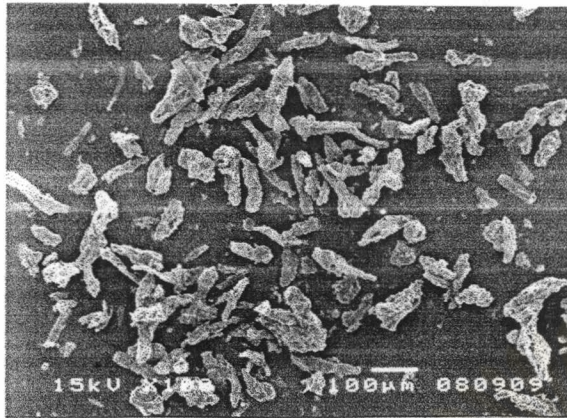
Topics	Avicel®101 <sup>1</sup>	Vivapur®101 <sup>2</sup>	Vitacel® <sup>2</sup>
Processing Steps	Highly Purified Pulp (Alpha Cellulose) ↓ Acid Hydrolysis ↓ Purification ↓ Attrition ↓ Spray Drying	Cellulose Pulp ↓ Precutting / Milling ↓ Acid hydrolysis ↓ Filtration ↓ Washing Process ↓ Neutralisation ↓ Washing Process ↓ Drying Process in Air Stream ↓ Sieving and Grinding Process ↓ Mixing and Packaging	Cellulose Pulp ↓ Precutting ↓ Grinding ↓ Sieving ↓ Packaging
Price (baht / kg)	360	120	About 70% of Vivapur®

Sources : 1 = Technical information sheet of FMC.

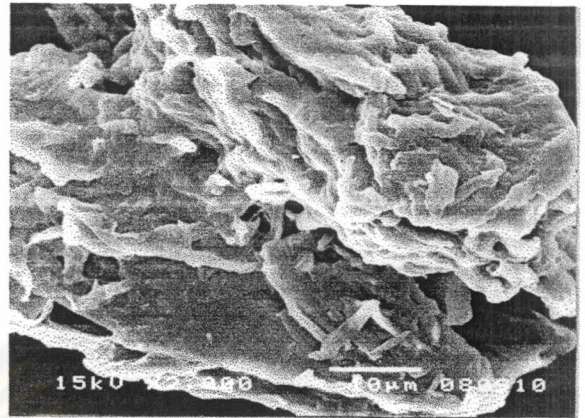
2 = Technical information sheet of JRS, March 1998 – 02.

hydrolysis treatment is used to remove amorphous part in cellulose to have the high content of crystalline part, which can form stronger hydrogen bonds than native cellulose which is composed of both amorphous and crystalline part. This bonding then give the high tablet strength and good disintegration (Czeisler and Perlman, 1991; Sixsmith, 1976). Disintegration times could be ranked in the order: Vitacel<sup>®</sup> tablets >>> Vivapur<sup>®</sup> tablets > Avicel<sup>®</sup> tablets. Vitacel<sup>®</sup> tablets gave the longest disintegration time due to the larger and fibrous shape particles (see Table 3-7 and Figure 3-2) which would interlock each other. Although it swelled and laminated rapidly in the medium, the tablets did not subsequently disintegrate and remained unchange until after 30 minutes. In the cases of Avicel<sup>®</sup> and Vivapur<sup>®</sup>, the disintegration time of Vivapur<sup>®</sup> was longer than that of Avicel<sup>®</sup>. By observation during disintegration test, Avicel<sup>®</sup> and Vivapur<sup>®</sup> tablets disintegrated rapidly into small pieces when contacting with water, but only disintegrated particles of Avicel<sup>®</sup> subsequently broken into smaller particle size and passed through the basket screen of disintegration test apparatus (sieve no. 10, diameter 2.00 mm, USPXXIII, 1995). Therefore, short disintegration times of Avicel<sup>®</sup> tablets were obtained. This was explained by the difference in the drying process of manufacturing procedure and their particle sizes as indicated in Table 3-6 and 3-7, respectively. Drying by spray drying technique (Avicel<sup>®</sup>) gives higher porous particles that can break more easily than air stream drying (Vivapur<sup>®</sup>) which produces more compacted particle as shown by SEM photomicrographs in Figure 3-2.

From the physical properties and the price, Vivapur<sup>®</sup> was chosen in the preparation of composite particles with starch. The particle sizes of the starting materials are shown in Table 3-8. The particle size of Vivapur<sup>®</sup> is larger than that of starch and may cause clogging problem in the nozzle of the rotary atomizer. Vivapur<sup>®</sup> was then reduced in size and classified into two categories, which were VS and VJM. VS was prepared by passing Vivapur<sup>®</sup> through sieve number 325 (45  $\mu\text{m}$  aperture) and VJM was prepared by reduction particle size of Vivapur<sup>®</sup> with jet milling. Size reduction by jet milling was performed three times and designated as VJM-1, VJM-2, and VJM-3. Their particle sizes are shown in Table 3-8 and Figure 3-3. It can be seen that the particle size of VJM was the smallest ( $D_{50} = 13.61$  to  $17.31 \mu\text{m}$ ) then that of VS ( $D_{50} = 40.51 \mu\text{m}$ ), and



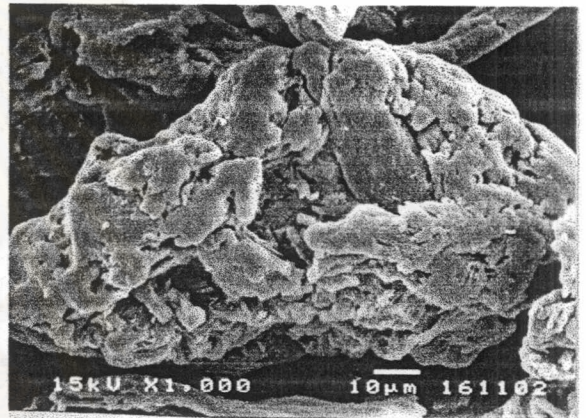
Avicel® 101 X 100



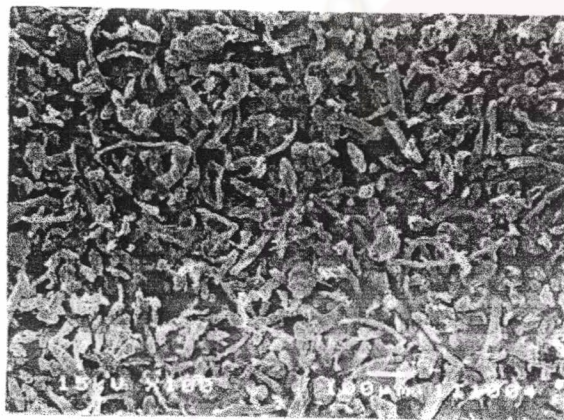
Avicel® 101 X 2000



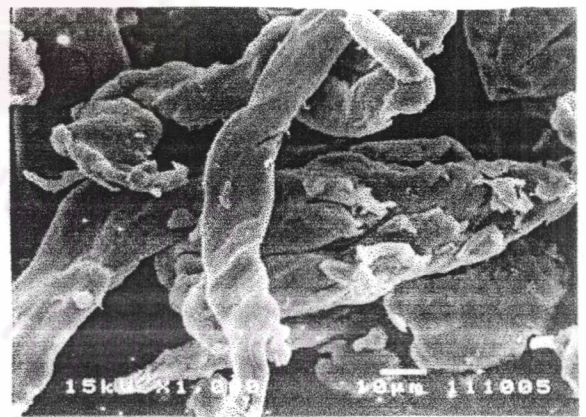
Vivapur® 101 X 100



Vivapur® 101 X 1000



Vitacel® X 100



Vitacel® X 1000

Figure 3-2 SEM photomicrographs of Avicel®101, Vivapur®101, and Vitacel®.

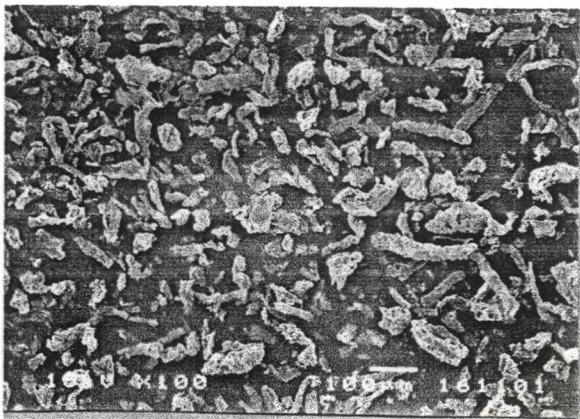
Table 3-7 Particle size distribution of MCC and powder cellulose.

MCC or Powder Cellulose	D(v, 0.1) ( $\mu\text{m}$ ) average (SD)	D(v, 0.5) ( $\mu\text{m}$ ) average (SD)	D(v, 0.9) ( $\mu\text{m}$ ) average (SD)	Span (D90-D10)/D50 average (SD)
Avicel <sup>®</sup> 101	6.75 (0.07)	31.16 (0.27)	81.65 (0.98)	2.40 (0.01)
Vivapur <sup>®</sup> 101	24.25 (0.12)	69.05 (0.91)	142.77 (5.17)	1.72 (0.05)
Vitacel <sup>®</sup>	24.20 (0.18)	75.53 (0.68)	205.55 (1.70)	2.40 (0.02)

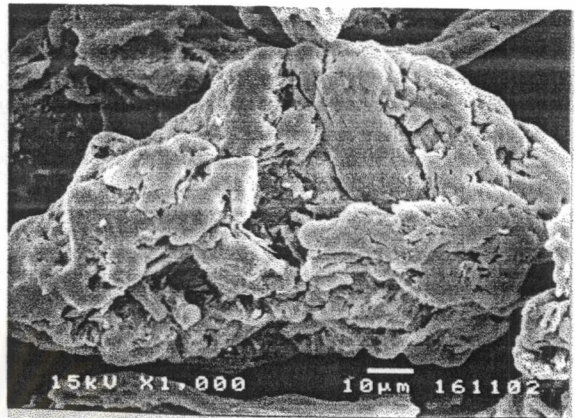
Table 3-8 Particle size distribution of RF, RS, and MCC.

Raw Materials	D(v, 0.1) ( $\mu\text{m}$ ) average (SD)	D(v, 0.5) ( $\mu\text{m}$ ) average (SD)	D(v, 0.9) ( $\mu\text{m}$ ) average (SD)	Span (D90-D10)/D50 average (SD)
RF	5.03 (0.38)	46.22 (0.56)	137.66 (0.96)	2.87 (0.06)
RS	0.56 (0.01)	13.57 (0.11)	77.51 (2.16)	5.67 (0.11)
Vivapur <sup>®</sup> 101	24.25 (0.12)	69.05 (0.91)	142.77 (5.17)	1.72 (0.05)
VS	12.92 (0.11)	40.51 (0.08)	86.90 (0.47)	1.83 (0.02)
VJM-1*	3.46 (0.13)	17.31 (0.17)	47.74 (2.44)	2.56 (0.12)
VJM-2	2.98 (0.11)	13.61 (0.51)	33.84 (1.98)	2.27 (0.05)
VJM-3	3.46 (0.05)	16.28 (0.08)	43.72 (1.03)	2.47 (0.08)

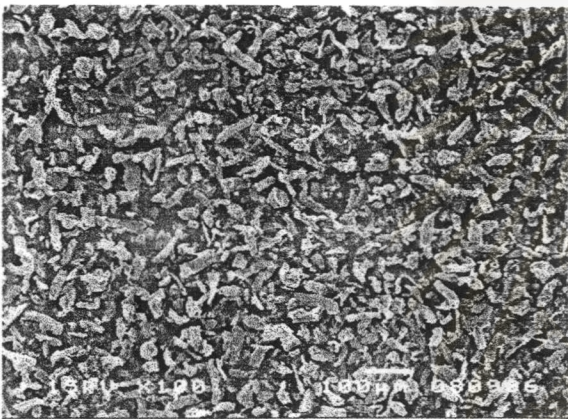
Note : \* = The number after hyphen indicates lot number of size reduction by jet milling e.g. VJM-1 is the first lot of production.



Vivapur® 101 X 100



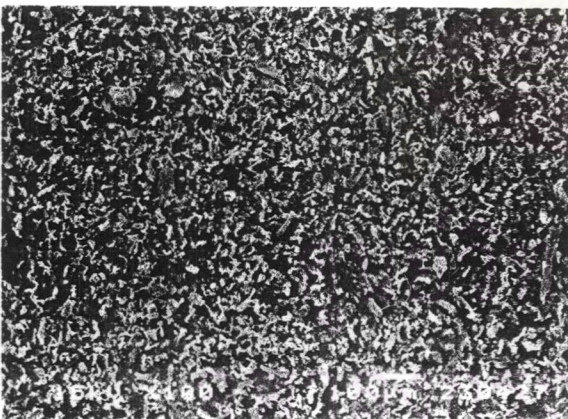
Vivapur® 101 X 1000



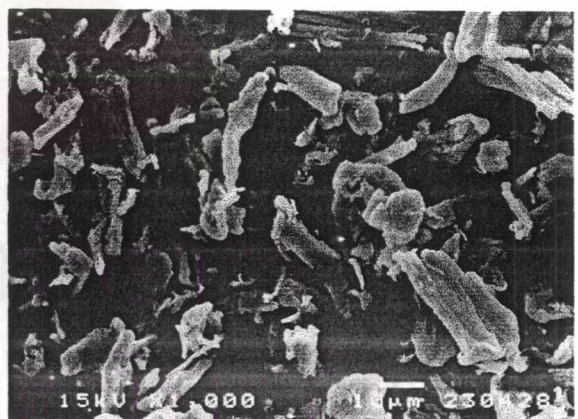
VS X 100



VS X 2000



VJM X 100



VJM X 1000

Figure 3-3 SEM photomicrographs of Vivapur® 101, VS, and VJM.

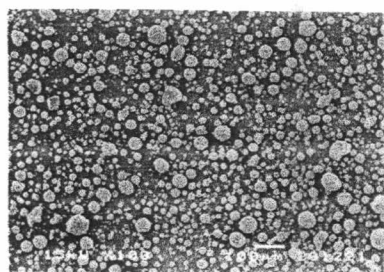
the largest size was that of Vivapur<sup>®</sup> ( $D_{50} = 69.05\mu\text{m}$ ). However, it was found that the particle sizes of Vivapur<sup>®</sup> after jet milling from three lots were similar.

### **Rice Flour and MCC Formulations**

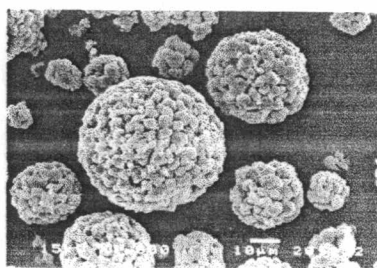
SEM photomicrographs of formulations are depicted in Figures 3-4 and 3-5. Spray-dried rice flour without MCC (RFSD) was in spherical form with various sizes when compared to RF which mostly in the aggregate form with irregular shape. With increase in the quantity of MCC in the preparation, not only did the larger size of the particles obtain, but also the more irregular shape was found. Comparing between composite particles rice flour and VS formulations, and composite particles rice flour and VJM formulations (RFVS and RFVJM formulations), RFVJM formulations gave more spherical shape of the spray-dried particles than RFVS formulations. Therefore, the difference in particle shape of spray-dried powder was due to the difference in particle size of MCC used. However, when MCC amount was increased, the spherical shape seemed to change, and many of MCC fibers on the particle surface could be seen at high magnification of SEM.

Percent yield and % LOD of rice flour (RF) and spray-dried powders of various formulations are presented in Table 3-9. By observation during preparation of spray drying suspension, an increase in MCC quantity in the preparation increased the viscosity of the suspension. This might be due to the hydration property of MCC, which bound the water around its fiber. The formulation with higher viscosity would give larger atomizing droplets and did not dry immediately when contacting with hot air during spray drying process. The droplets then adhered to the wall of the spray dryer chamber. Therefore, the higher proportion of MCC in composite particles was, the less % yield was obtained. At the same ratio of rice flour and MCC, % yields of RFVS formulations were slightly greater than that of RFVJM formulations.

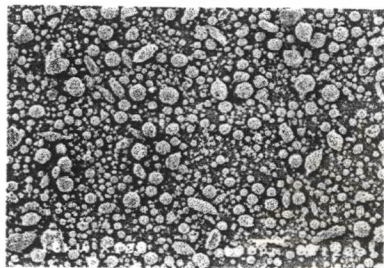
Physical properties of tablets made from composite particles of RF and MCC formulations are shown in Table 3-10. It was found that RF without modification gave the poorest hardness, and all tablets were broken when subjected to friability test. VS and VJM gave the highest hardness values, which could not be measured by the hardness



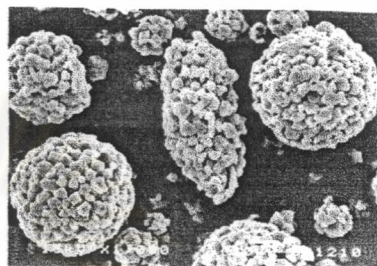
RFSD X 100



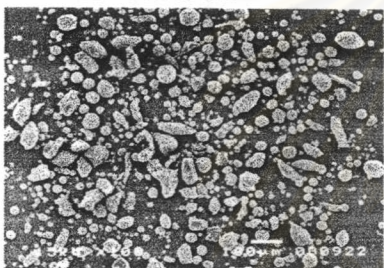
RFSD X 1000



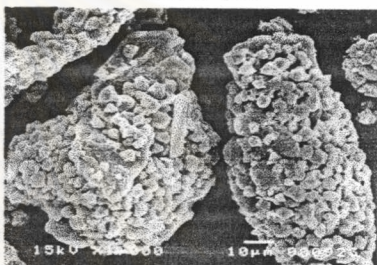
RFVS-91 X 100



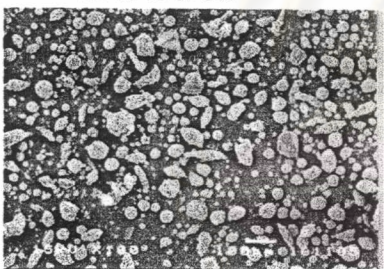
RFVS-91 X 1000



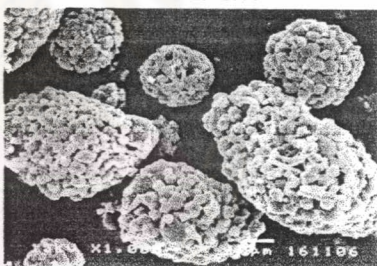
RFVS-82 X 100



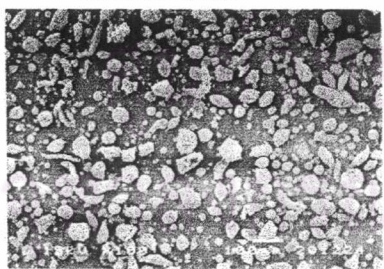
RFVS-82 X 1000



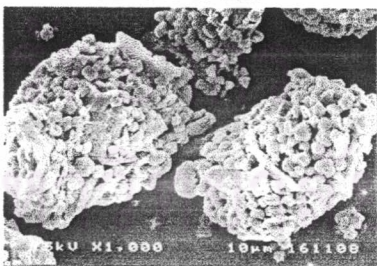
RFVS-73 X 100



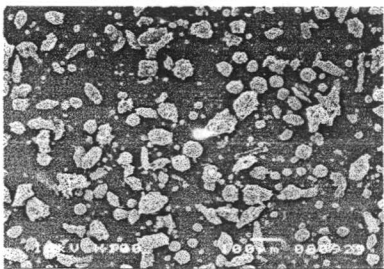
RFVS-73 X 1000



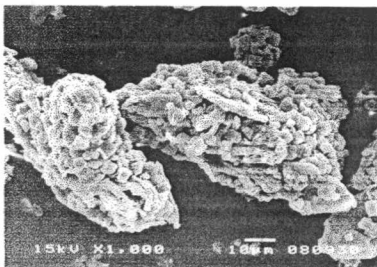
RFVS-64 X 100



RFVS-64 X 1000



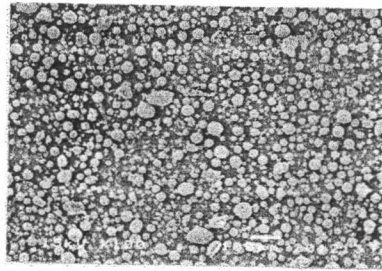
RFVS-55 X 100



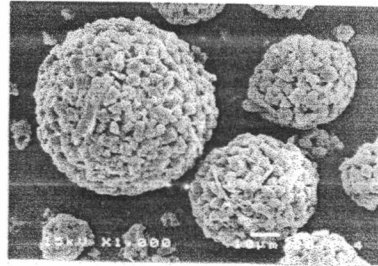
RFVS-55 X 1000

Figure 3-4 SEM photomicrographs of RFSD, RFVS-91 to RFVS-55.





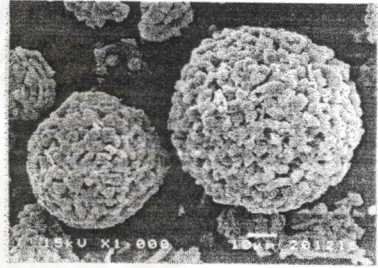
RFVJM-91 X 100



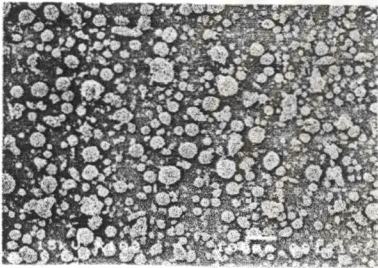
RFVJM-91 X 1000



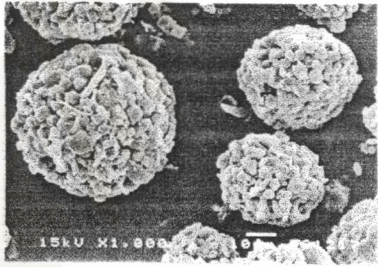
RFVJM-82 X 100



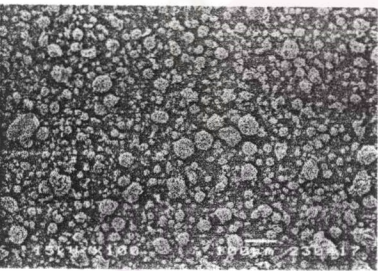
RFVJM-82 X 1000



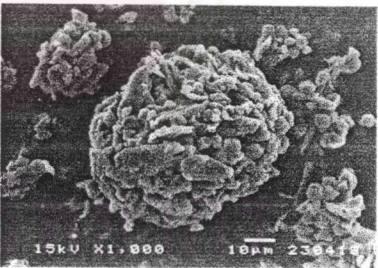
RFVJM-73 X 100



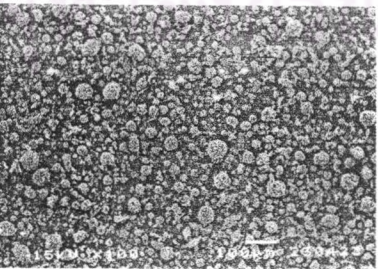
RFVJM-73 X 1000



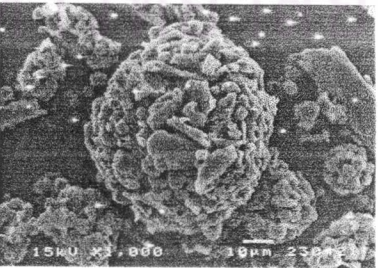
RFVJM-64 X 100



RFVJM-64 X 1000



RFVJM-55 X 100



RFVJM-55 X 1000

Figure 3-5 SEM photomicrographs of RFVJM-91 to RFVJM-55.

Table 3-9 Physical properties of powder of RF and spray dried formulations of RF and MCC.

Materials	Yield (%)	LOD (%) average (SD)
RF	NA	11.33 (0.13)
RF dried	NA	6.65 (0.46)
VS	NA	5.64 (0.26)
VJM	NA	5.33 (0.05)
RFSD	77.23	5.11 (0.07)
RFVS-91	64.22	4.73 (0.14)
RFVS-82	63.37	5.27 (0.08)
RFVS-73	58.26	4.31 (0.05)
RFVS-64	52.62	3.98 (0.19)
RFVS-55	51.44	4.57 (0.13)
RFVJM-91	65.36	4.40 (0.11)
RFVJM-82	57.78	4.70 (0.03)
RFVJM-73	50.42	4.75 (0.23)
RFVJM-64	49.84	5.29 (0.15)
RFVJM-55	45.91	4.86 (0.15)

Note : NA = not applicable

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Table 3-10 Tablet properties of composite particles of RF and MCC formulations prepared by hydraulic press at compression force of 2000 lb.

Formulations	Hardness (N) average (SD)	Diameter (mm) average (SD)	Thickness (mm) average (SD)	Friability (%)	DT (min) average (SD)
RF	22.5 (2.26)	12.86 (0.01)	3.34 (0.03)	all tablets were broken	0.96 (0.09)
VS	--*	12.83 (0.01)	3.26 (0.02)	0.00	>30
VJM	--*	12.82 (0.04)	3.24 (0.02)	0.00	>30
RFSD	22.2 (2.26)	12.89 (0.02)	3.49 (0.03)	all tablets were broken	1.13 (0.09)
RFVS-91	31.3 (3.34)	12.89 (0.02)	3.52 (0.03)	all tablets were broken	0.82 (0.09)
RFVS-82	54.2 (4.81)	12.89 (0.02)	3.51 (0.03)	2.67	0.61 (0.08)
RFVS-73	78.0 (5.20)	12.85 (0.01)	3.47 (0.03)	1.39	0.50 (0.11)
RFVS-64	91.8 (3.63)	12.89 (0.01)	3.47 (0.02)	0.90	0.54 (0.08)
RFVS-55	122.4 (2.45)	12.89 (0.01)	3.40 (0.10)	0.50	0.34 (0.09)
RFVJM-91	35.4 (2.35)	12.90 (0.02)	3.54 (0.02)	3.09	0.86 (0.12)
RFVJM-82	60.9 (3.04)	12.88 (0.01)	3.49 (0.03)	1.70	0.60 (0.05)
RFVJM-73	70.2 (4.22)	12.88 (0.03)	3.49 (0.05)	1.29	0.52 (0.08)
RFVJM-64	93.2 (9.81)	12.87 (0.03)	3.48 (0.06)	1.09	0.51 (0.11)
RFVJM-55	124.2 (9.32)	12.86 (0.02)	3.48 (0.06)	0.50	0.40 (0.09)

Note : \* = very hard tablets and could not be measured by hardness tester

tester. RFSD gave the same hardness as RF. For composite particles of RF and MCC formulations, as the quantity of MCC was increased, the hardness of the tablets was also increased while the disintegration time was decreased. This was due to the compressibility and disintegration properties of MCC. RFVS and RFVJM formulations at the same ratio gave similar results in the tablet properties, e. g. hardness, % friability, and disintegration time. Percent friability and disintegration time of RFVS-64, RFVS-55 and RFVJM-55 were less than 1% and less than 1 minute, respectively. From the results above, spray drying process could not improve compressibility of rice flour, but the incorporation of MCC produced increased hardness of tablets. It could be explained by the highly compressible property of MCC. The higher quantity of MCC in the preparations gave higher compressibility, and then higher hardness was obtained.

Although the higher proportion of MCC gave higher hardness, but the satisfactory results of the tablets that had hardness with lower % friability (less than 1 %) and minimum disintegration time were obtained in the formulations of RFVS-64, RFVS-55, and RFVJM-55. However, the high percentage of MCC was not preferred because of the higher cost of starting materials.

#### **Rice Starch and MCC Formulations**

Scanning electron photomicrographs of spray dried rice starch without MCC (RSSD) and composite particles of RS and MCC formulations are depicted in Figures 3-6 and 3-7. RSSD was in spherical form with welding of starch grains at contact points could be seen at higher magnification. In RSVS formulations, increase in MCC quantity resulted in rugby-ball like particles, and irregular shape particles were found at the MCC amount higher than 30%. This was due to long fibers of VS that had greater size than starch grain. In the case of RSVJM formulations, the sphericity of particles was at higher degree than that of RSVS formulations. This is because VJM particles had a smaller size than VS. However, an increase in MCC proportion in the formulation resulted in the reduction of the spherical form and increased irregularity of the surface of the particles.

Physical properties of RS, VS, VJM and composite particles of RS and MCC formulations are shown in Table 3-11. Percent yield of RSSD was 60.58% while those of

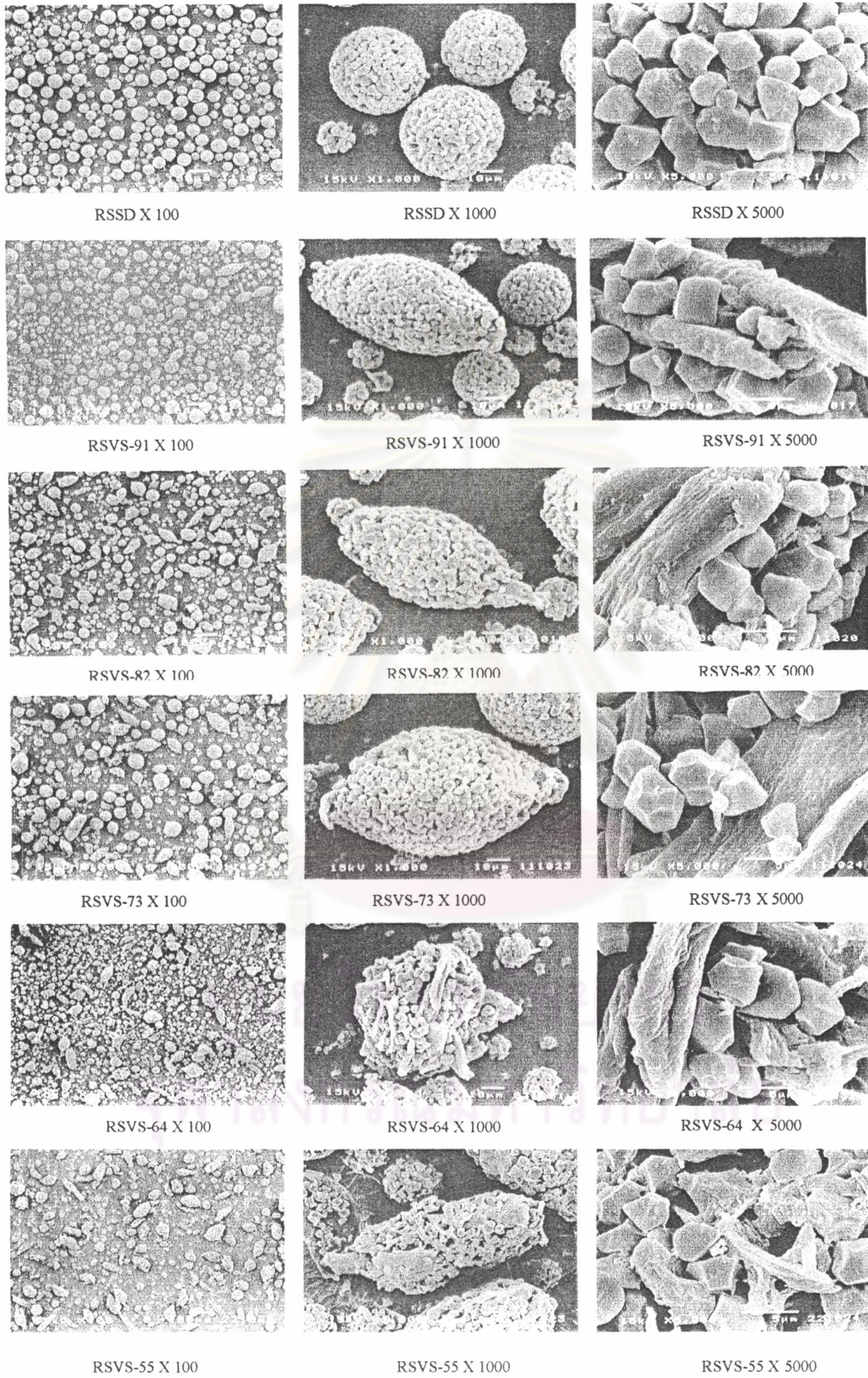


Figure 3-6 SEM photomicrographs of RSSD and RSVS-91 to RSVS-55.

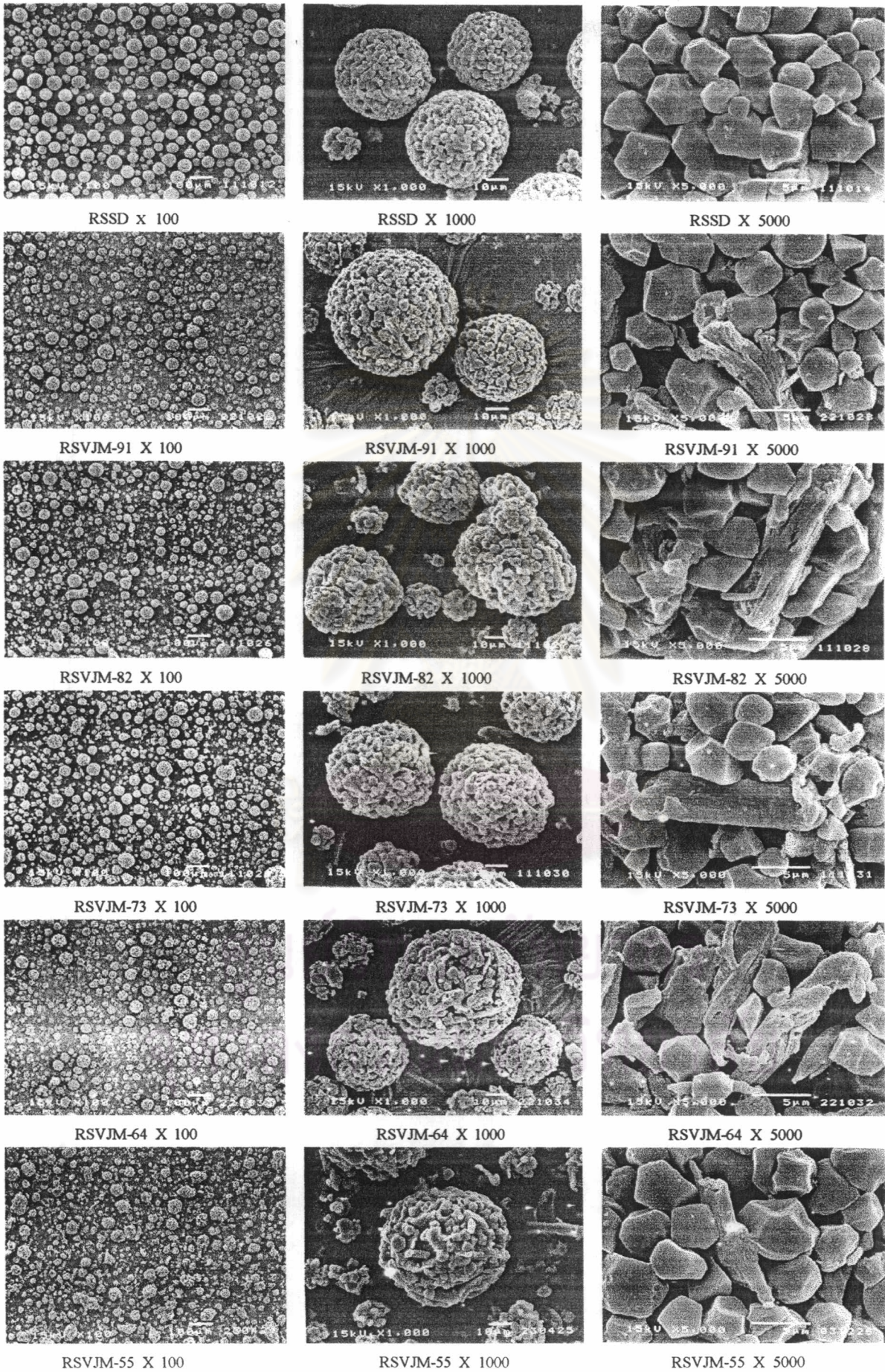


Figure 3-7 SEM photomicrographs of RSSD and RSVJM-91 to RSVJM-55.

Table 3-11 Physical properties of RS, RSSD, and spray dried formulations of RS and MCC.

Materials	Yield (%)	LOD (%) average (SD)	Angle of Repose (degree) average (SD)	Angle of Spatula (degree) average (SD)	Bulk Density (g/ml) average (SD)	Packed Density (g/ml) average (SD)	Compressibility (%) average (SD)	Cohesion (%) average (SD)	Flowability Index average (SD)
RS	NA	11.40 (0.07)	--*	--*	0.335 (0.02)	0.605 (0.01)	44.58 (3.39)	--*	1.3 (1.15)
VS	NA	5.64 (0.26)	46.3 (0.65)	66.3 (3.86)	0.264 (0.00)	0.427 (0.00)	38.30 (0.49)	0.5 (0.01)	45.2 (1.44)
RSSD	60.58	6.77 (0.06)	30.8 (0.51)	55.6 (2.58)	0.534 (0.00)	0.598 (0.00)	10.71 (0.26)	14.4 (0.00)	72.3 (0.29)
RSVS-91	59.99	6.61 (0.14)	29.8 (0.76)	55.6 (2.95)	0.501 (0.00)	0.582 (0.01)	13.90 (1.08)	8.8 (0.00)	74.0 (1.50)
RSVS-82	61.28	6.34 (0.10)	31.1 (0.25)	60.8 (0.96)	0.476 (0.00)	0.581 (0.01)	18.11 (0.92)	7.3 (0.00)	68.3 (1.61)
RSVS-73	49.22	5.88 (0.26)	36.4 (1.43)	62.1 (3.65)	0.434 (0.01)	0.536 (0.01)	19.15 (0.47)	3.4 (0.00)	65.3 (2.84)
RSVS-64	58.60	5.35 (0.34)	36.0 (1.81)	66.2 (2.01)	0.417 (0.00)	0.524 (0.00)	20.47 (0.44)	3.4 (0.00)	63.5 (1.32)
RSVS-55	51.14	4.67 (0.06)	35.8 (0.90)	67.3 (3.65)	0.412 (0.00)	0.524 (0.00)	21.42 (0.66)	4.9 (0.00)	63.3 (0.76)
RSVJM-91	61.44	6.88 (0.05)	29.4 (1.75)	58.5 (2.25)	0.478 (0.00)	0.560 (0.01)	14.70 (0.54)	9.3 (0.00)	73.5 (1.73)
RSVJM-82	50.62	6.40 (0.21)	32.9 (0.36)	61.8 (2.98)	0.436 (0.00)	0.532 (0.01)	17.97 (1.32)	8.4 (0.01)	67.7 (2.02)
RSVJM-73	49.03	5.50 (0.08)	37.2 (2.15)	57.9 (2.08)	0.426 (0.01)	0.527 (0.00)	19.23 (1.56)	3.2 (0.00)	67.0 (1.73)
RSVJM-64	46.79	5.69 (0.09)	36.9 (0.06)	62.7 (2.69)	0.365 (0.01)	0.462 (0.01)	20.99 (1.40)	4.4 (0.01)	63.3 (2.25)
RSVJM-55	38.98	4.90 (0.19)	39.4 (0.69)	66.6 (2.52)	0.352 (0.01)	0.464 (0.01)	24.24 (1.95)	4.7 (0.02)	59.5 (1.73)

Note : NA = not applicable

\* = cannot be determined

composite particles of RS and MCC formulations were lower. An increase in MCC content, a lower in % yield was obtained. This was due to the adhesion of spray dried droplets to the chamber wall. Native RS gave the highest moisture value while % LOD of all spray-dried powders was lower and around 4.67 to 6.88%. Flow properties of starting materials and all spray-dried powders were evaluated in terms of angle of repose, % compressibility, and flowability index. Angle of repose of native RS could not be determined because plugging occurred during the measurement and VS showed the highest value, which indicated poor flowability. An increase of MCC proportion in the RS and MCC formulations was, the higher angle of repose was obtained. The higher value of repose angle indicates the lower flowability of materials. Then increase in MCC content in composite particles decreased the flowability of powder. Higher percent compressibility and lower flowability index of composite particles formulations were observed with increased the MCC proportion. Higher percent compressibility and lower flowability index indicate less flowability. According to the values of angle of repose, % compressibility, and flowability index, the predicted flowability of powder might be ranked in the order of RSSD > RSVS or RSVJM at low MCC proportion > RSVS or RSVJM at high MCC proportion > VS > RS. These could explain by particle size, particle shape, and percent LOD of powders. RS was composed of small starch grain that aggregated in irregular shape, some was in single particle and also had the high moisture content which possessed poor flowability. The fibrous-shape like of VS could resist the flow. RSSD was in spherical form, which exhibited the highest flowability. When the amount of MCC was increased in the composite particles of RS and MCC formulations, the more irregularity of the particles was observed. This would lead to the decrease in flowability of the powder.

Tablet properties of RS and MCC formulations are presented in Table 3-12. VS and VJM gave the highest hardness. RS and RSSD tablets presented the higher hardness than RF and RFSD tablets. As in the case of RF and RFSD, spray drying process only improved the flowability of powder by transformation of the particles in spherical form, but did not affect the hardness of resultant tablets. The difference in the hardness of RF and RS tablets was from the different amounts of protein content in the powder.



Table 3-12 Physical properties of tablets of spray-dried formulations of RS and MCC prepared by hydraulic press at compression force of 2000 lb.

Materials	Hardness (N) average (SD)	Diameter (mm) average (SD)	Thickness (mm) average (SD)	Friability (%)	DT (min) average (SD)
RS	131.4 (8.83)	12.87 (0.02)	3.46 (0.04)	0.60	1.88 (0.09)
VS	--*	12.83 (0.01)	3.26 (0.02)	0.00	>30
VJM	--*	12.82 (0.04)	3.24 (0.02)	0.00	>30
RSSD	128.2 (15.99)	12.89 (0.04)	3.60 (0.05)	0.77	2.06 (0.15)
RSVS-91	160.6 (11.28)	12.83 (0.02)	3.56 (0.07)	0.59	2.47 (0.21)
RSVS-82	164.6 (18.74)	12.86 (0.04)	3.58 (0.06)	0.40	3.16 (0.25)
RSVS-73	189.8 (10.59)	12.87 (0.03)	3.50 (0.04)	0.30	2.77 (0.34)
RSVS-64	201.2 (7.06)	12.87 (0.02)	3.46 (0.05)	0.20	2.40 (0.46)
RSVS-55	228.0 (10.00)	12.85 (0.01)	3.49 (0.04)	0.10	0.92 (0.15)
RSVJM-91	141.5 (10.69)	12.87 (0.02)	3.63 (0.06)	0.69	2.80 (0.21)
RSVJM-82	172.2 (14.52)	12.90 (0.04)	3.57 (0.07)	0.40	3.04 (0.24)
RSVJM-73	188.7 (8.63)	12.89 (0.03)	3.47 (0.06)	0.32	2.56 (0.22)
RSVJM-64	220.8 (14.42)	12.86 (0.02)	3.48 (0.03)	0.10	2.07 (0.76)
RSVJM-55	229.6 (12.75)	12.87 (0.02)	3.48 (0.03)	0.20	1.09 (0.13)

Note : \* = very hard tablets and could not be measured by hardness tester

Since protein has many different functional groups such as carboxy and amino groups, these groups can interact with hydroxy group of starch in some way (Timaroon, 1994; Weecharangsan, 1995). Therefore, hydrogen bonding occurred during the compression could be reduced or inhibited and resulted in lower hardness of RF tablets than that of RS tablets. When MCC was incorporated in the formulations, an increase in the quantity of MCC would increase tablet hardness and decrease % friability and disintegration time. This is due to the high compressibility of MCC, which was added in the formulations and produced the higher hardness. MCC also behaves as disintegrant when used in the range 5 to 15% (Kibbe, 2000), thus disintegration time was reduced despite the higher hardness of tablets. This result was in contrary to a previous study where physical combinations of modified rice starch and MCC at ratio of 1 : 1 resulted in poor flowability of the blends and slow disintegration time of the tablets resulted (Bos et al., 1992). But the composite particles produced by spray drying technique in this research possessed the high compressibility, good flowability and disintegration, this made it is suitable to be used as directly compressible diluent.

In order to select the formulation for further study, the physical properties and the amount of MCC in the formulation should be considered. All formulations gave good results of disintegration time and % friability that were less than 5 minutes and less than 1%, respectively. Although an increase in the MCC enhanced tablets hardness, higher amount of MCC was not preferred due to the higher cost of starting material. The formulation of RS and MCC at ratio of 7 : 3 was selected because it gave suitable results of hardness with appropriate amount of MCC. Moreover, commercial coprocessed excipient with cellulose is usually comprised of cellulose by about 30%.

The powder properties of various DC diluents and selected composite particles are shown in Table 3-13. Flowability of composite particles in comparison with various DC diluents is as follows : Eratab<sup>®</sup> > RSVJM-73 ≈ RSVS-73 > Tablettose<sup>®</sup> ≈ Cellactose<sup>®</sup> > Vivapur<sup>®</sup>. Eratab<sup>®</sup> and composite particles of RS and MCC gave higher flowability than other DC diluents because of the more spherical form of the particles. Tablet properties of various DC diluents are tabulated in Table 3-14. The tablets hardness could be ranked in the following order: Vivapur<sup>®</sup> >> RSVS-73 ~ RSVJM-73 > Eratab<sup>®</sup> >

Table 3-13 Physical properties of powder of RSVS-73, RSVJM-73, and various DC diluents.

Materials	Angle of Repose (degree) average (SD)	Angle of Spatula (degree) average (SD)	Bulk Density (g/ml) average (SD)	Packed Density (g/ml) average (SD)	Compressibility (%) average (SD)	Cohesion (%) average (SD)	Flowability Index average (SD)
RSVS-73	36.4 (1.43)	62.1 (3.65)	0.434 (0.01)	0.536 (0.01)	19.15 (0.47)	3.4 (0.00)	65.3 (2.84)
RSVJM-73	37.2 (2.15)	57.9 (2.08)	0.426 (0.01)	0.527 (0.00)	19.23 (1.56)	3.2 (0.00)	67.0 (1.73)
Vivapur <sup>®</sup> 101	39.5 (0.75)	64.7 (1.27)	0.298 (0.00)	0.442 (0.00)	32.58 (0.60)	0.0 (0.01)	52.5 (1.73)
Eratab <sup>®</sup>	33.2 (1.76)	55.5 (2.90)	0.543 (0.00)	0.624 (0.00)	13.03 (0.40)	12.3 (0.00)	70.0 (0.00)
Tabletose <sup>®</sup>	36.6 (1.07)	59.3 (1.79)	0.549 (0.00)	0.718 (0.00)	23.50 (0.22)	47.2 (0.01)	57.2 (1.76)
Cellactose <sup>®</sup>	39.6 (1.59)	57.3 (2.04)	0.414 (0.00)	0.528 (0.00)	21.53 (0.50)	45.3 (0.00)	57.0 (0.00)

Table 3-14 Physical properties of tablets of RSVS-73, RSVJM-73, and various DC diluents prepared by hydraulic press at compression force of 2000 lb.

Materials	Hardness (N) average (SD)	Diameter (mm) average (SD)	Thickness (mm) average (SD)	Friability (%)	DT (min) average (SD)
RSVS-73	189.8 (10.59)	12.87 (0.03)	3.50 (0.04)	0.30	2.77 (0.34)
RSVJM-73	188.7 (8.63)	12.89 (0.03)	3.47 (0.06)	0.32	2.56 (0.22)
Vivapur <sup>®</sup> 101	--*	12.81 (0.01)	3.61 (0.03)	0.00	> 30
Eratab <sup>®</sup>	131.6 (11.18)	12.86 (0.03)	3.34 (0.04)	1.42	2.11 (0.05)
Tabletose <sup>®</sup>	30.9 (1.67)	12.78 (0.02)	3.12 (0.03)	--**	--***
Cellactose <sup>®</sup>	72.2 (4.22)	12.83 (0.01)	3.30 (0.01)	1.20	0.31 (0.06)

Note : \* = could not be measured by hardness tester

\*\* = all tablets were broken after testing

\*\*\* = not determined

Cellactose<sup>®</sup> > Tablettose<sup>®</sup>. Vivapur<sup>®</sup> gave the highest hardness tablets due to the high compressibility of this material, however, the disintegration time was longer than 30 minutes. Because RSVS-73 and RSVJM-73 are composite particles of rice starch and MCC, they gave higher hardness than Eratab<sup>®</sup>, which was modified rice starch without cellulose. Cellactose<sup>®</sup> gave the higher tablet hardness than Tablettose<sup>®</sup> because Cellactose<sup>®</sup> is spray dried mixture of 75 parts of lactose monohydrate and 25 parts of cellulose powder while Tablettose<sup>®</sup> is aggregate lactose composed of only lactose monohydrate. When composite particles of RS and MCC was compared with Cellactose<sup>®</sup>, both having the similar percentage of cellulose content, it gave tablets with higher hardness than Cellactose<sup>®</sup>. Although powder and tablet properties of RSVJM-73 and RSVS-73 were similar, only RSVJM-73 was selected for further study. The reason was the more spherical form of particles when using jet milled MCC in the formulation and particle size reduction by jet mill is more effective and suitable for industrial scale than VS which was obtained by sieving through mechanical sieve shaker. Moreover, % yield (determined by weighing the quantity of MCC before and after size reduction) of VJM was higher than that of VS, at 79.48 % and 40.17 %, respectively (see appendix 1).

#### Physicochemical Properties of Powder

IR spectra of RS, RSSD, and Eratab<sup>®</sup> are shown in Figure 3-8. The peak appeared at  $3400\text{ cm}^{-1}$  resulted from OH-stretching. CH-stretching was seen at  $2900\text{ cm}^{-1}$ . Peaks between  $1100\text{-}1160\text{ cm}^{-1}$  result from CO-stretching of COH and COC groups. For characterization of carbohydrate, peaks between  $730\text{-}960\text{ cm}^{-1}$  were used to identify (Colthup, Daly and Wiberly, 1975; Newman et al., 1996). IR spectra of RS, RSSD, and Eratab<sup>®</sup> showed peaks at around  $766$ ,  $861$ , and  $930\text{ cm}^{-1}$ . This indicated that all three materials had the absorbance peaks that were consistent with the structure of starch. IR spectra of three materials are similar and coincident. This indicated no chemical changes when RS was subjected to spray drying. X-ray spectra of RS, RSSD, and Eratab<sup>®</sup> are shown in Figure 3-9. X-ray patterns of three materials were similar to A type pattern, which is found in starch obtained from cereal-starches (Newman et al., 1996; Hizukuri, 1996). Since there are no change in X-ray patterns of starch when subjected to spray

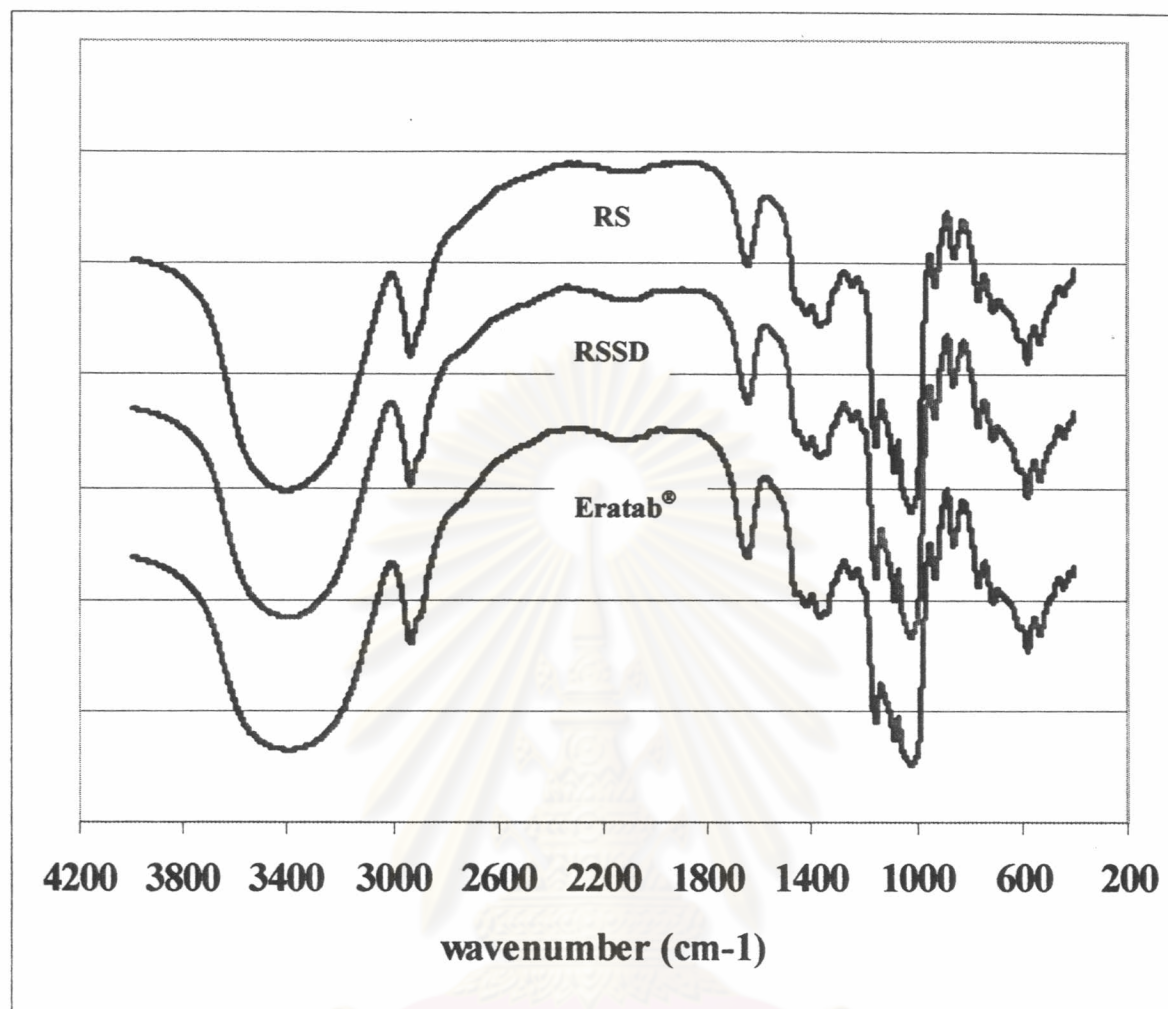


Figure 3-8 IR spectra of RS, RSSD, and Eratab®.

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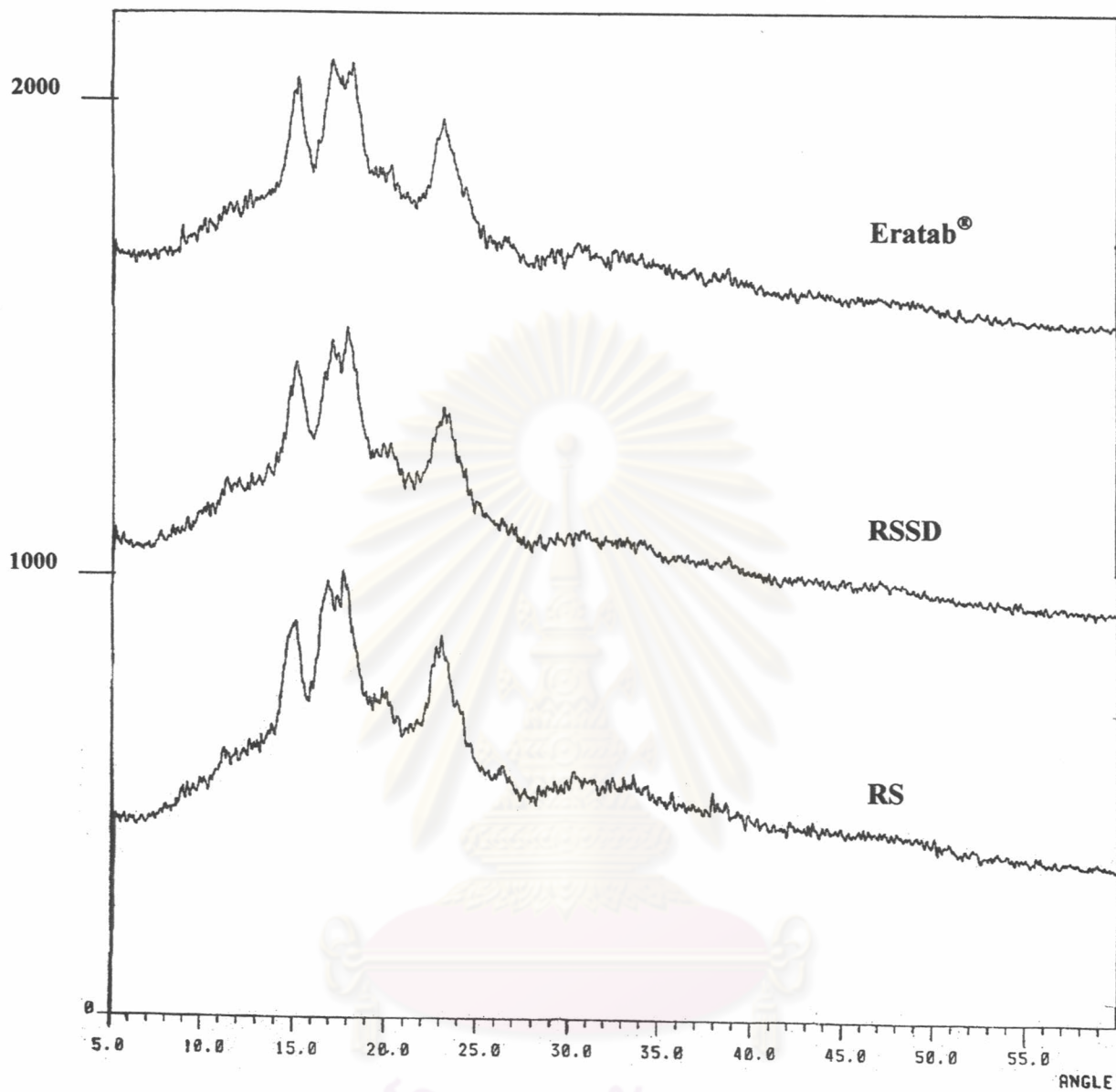


Figure 3-9 X-ray diffractograms of RS, RSSD, and Eratab<sup>®</sup>.

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drying, this indicated that RSSD and Eratab<sup>®</sup> have the internal molecular structure arrangement resemble to that of starting RS. Spray drying might produce gelatinization only at the surface of starch grain then the main crystalline granule structure was retained as native RS. DSC thermograms displayed in Figure 3-10 and showed endothermic peak at around 77.16° to 79.55°C, which would be the gelatinization temperature (Rutenberg, 1980). The enthalpy of gelatinization of RS, RSSD, and Eratab<sup>®</sup> is shown in Table 3-15. Enthalpy of gelatinization is used to measure degree of gelatinization (Wooton and Bamunuarachchi, 1979). Since these values were similar, degree of gelatinization of RSSD and Eratab<sup>®</sup> appeared unchanged. Although SEM showed fusion at contact point of starch grain surface, change of degree of gelatinization was not obtained by DSC. This meant that degree of gelatinization was rather low and/or could not be detected by DSC. This unchanged and/or rather little degree of gelatinization resulted in the same hardness of RS and RSSD tablets. DSC of composite particles of RS and MCC formulations are presented in Figure 3-11. VJM powder and VJM suspension showed little endothermic peak around 80 °C. The reason for which is not completely understood. However, it seems likely that endothermic peak might be due to water loss or differences in heat capacity between solid state of cellulose and the liquid state of cellulose. RS and RSVJM-91 to RSVJM-55 suspension gave endothermic peak around 74.80 ° to 77.43 °C. When MCC content was increased in the formulations, the decrease in the gelatinization enthalpy ( $\Delta H$ ) was shown (see Table 3-16). This indicated an increase in degree of gelatinization and might be explained by the hydration property of MCC in atomizing droplets, which would promote gelatinization during contact to the hot air. The higher MCC in the preparation, the more hydration in the droplet during spray drying and the higher degree of gelatinization was obtained. Due to the gelatinized granule in Starch<sup>®</sup> 1500 behave plastic deformation during compression, the improvement of compressibility was obtained. Therefore, this starch gelatinized would serve as a binder to improve the binding property accompanied with high compressibility of MCC in the formulations. IR spectrum and X-ray diffractogram of the selected formulation, RSVJM-73, were also investigated and shown in Figures 3-12 and 3-13, respectively. IR spectrum of RSVJM-73 resembles and superimposes to RS due to the high proportion of

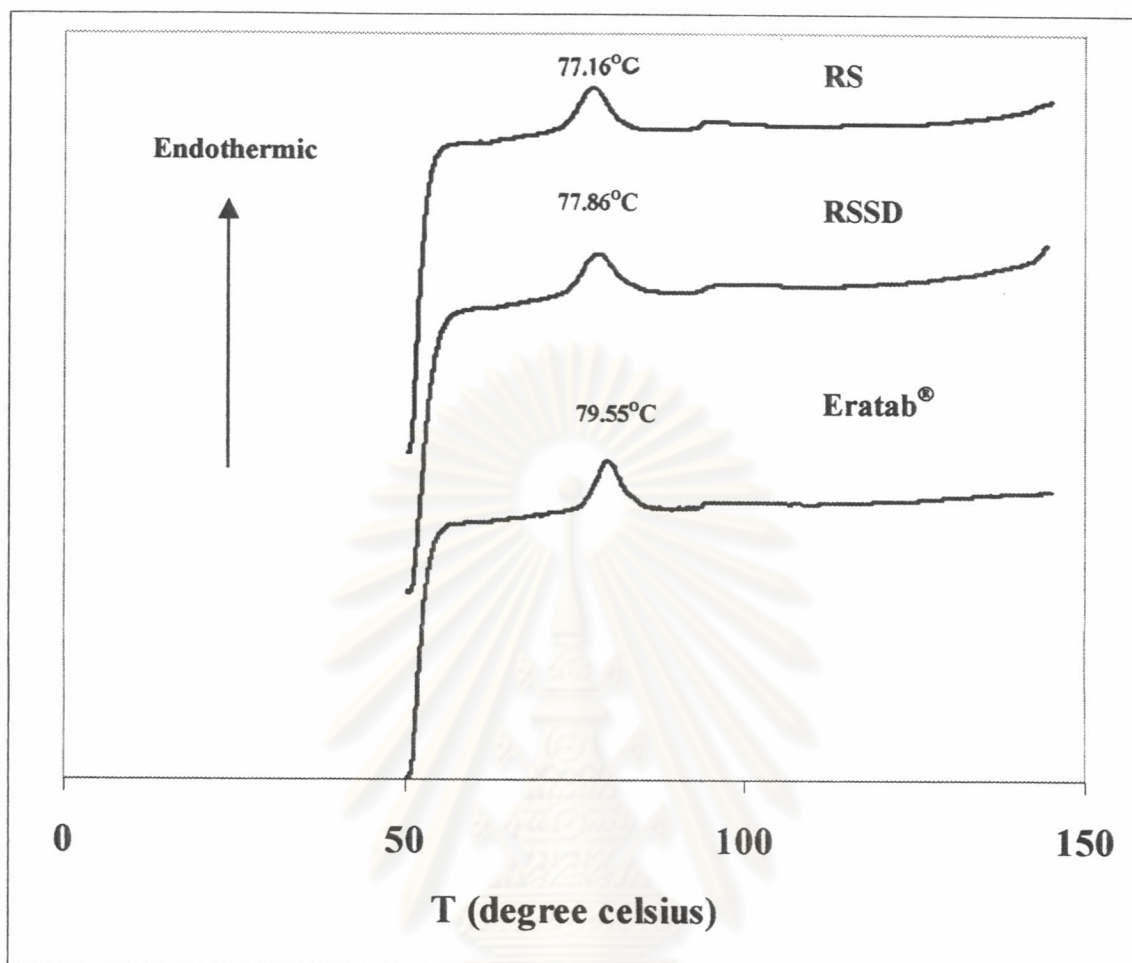


Figure 3-10 DSC thermograms of RS, RSSD, and Eratab® (approximately 20%w/w suspension).

Table 3-15 Endothermic properties from DSC thermograms of RS, RSSD, and Eratab®.

Materials	Endothermic Properties		
	$T_o$ (°C)	$T_p$ (°C)	$\Delta H$ (J/g)
RS	72.89	77.16	10.23
RSSD	73.71	77.86	10.49
Eratab®	76.10	79.55	10.91



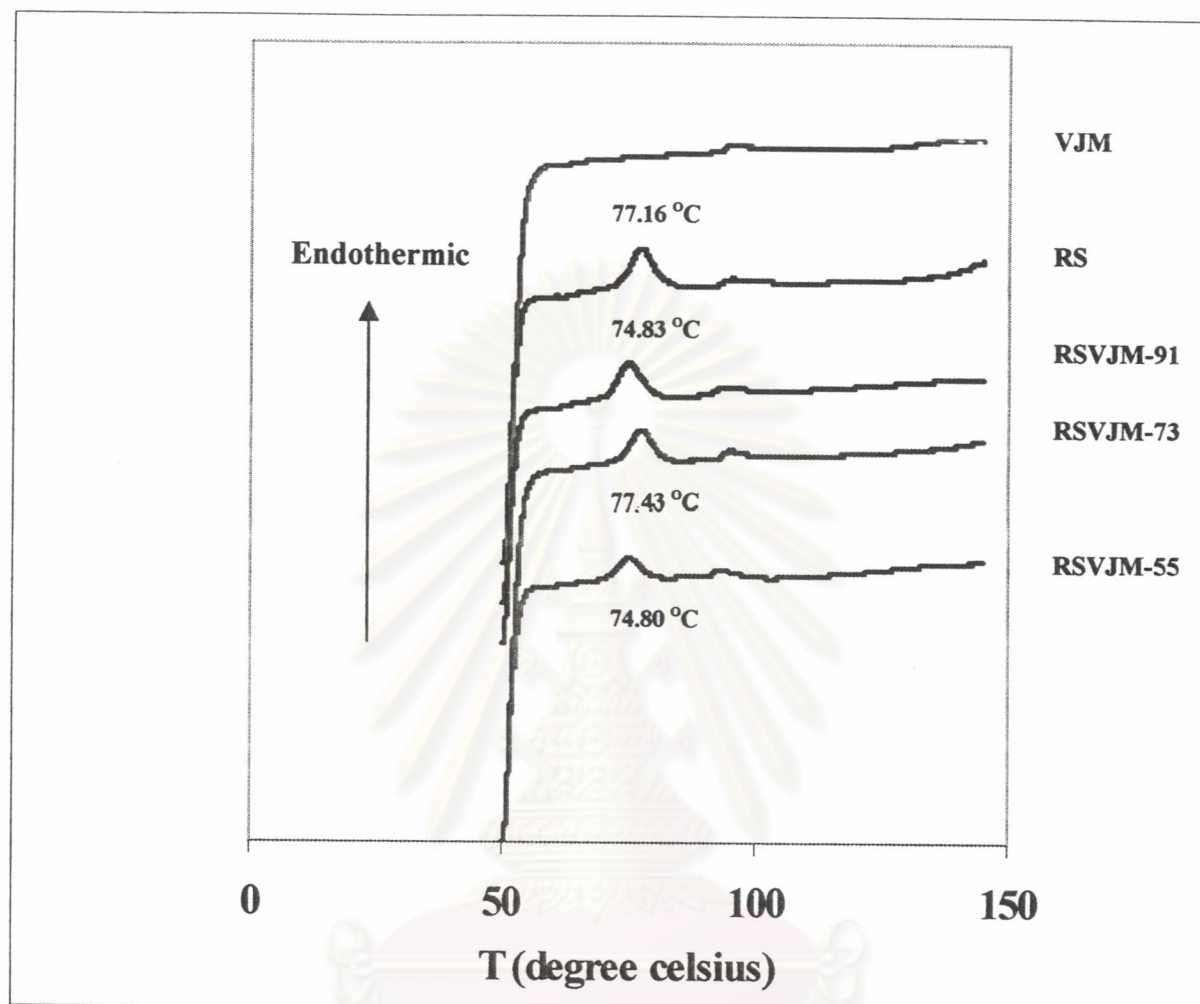


Figure 3-11 DSC thermograms of VJM, RS, RSVJM-91, RSVJM-73, and RSVJM-55.

Table 3-16 Endothermic properties from DSC thermograms of RS, RSSD, and composite particles of RS and MCC formulations.

Materials	Endothermic Properties		
	$T_o$ (°C)	$T_p$ (°C)	$\Delta H$ (J/g)
RS	72.89	77.16	10.23
RSSD	73.71	77.86	10.49
RSVJM-91	71.07	74.83	10.31
RSVJM-73	73.27	77.43	8.52
RSVJM-55	71.43	74.80	6.26

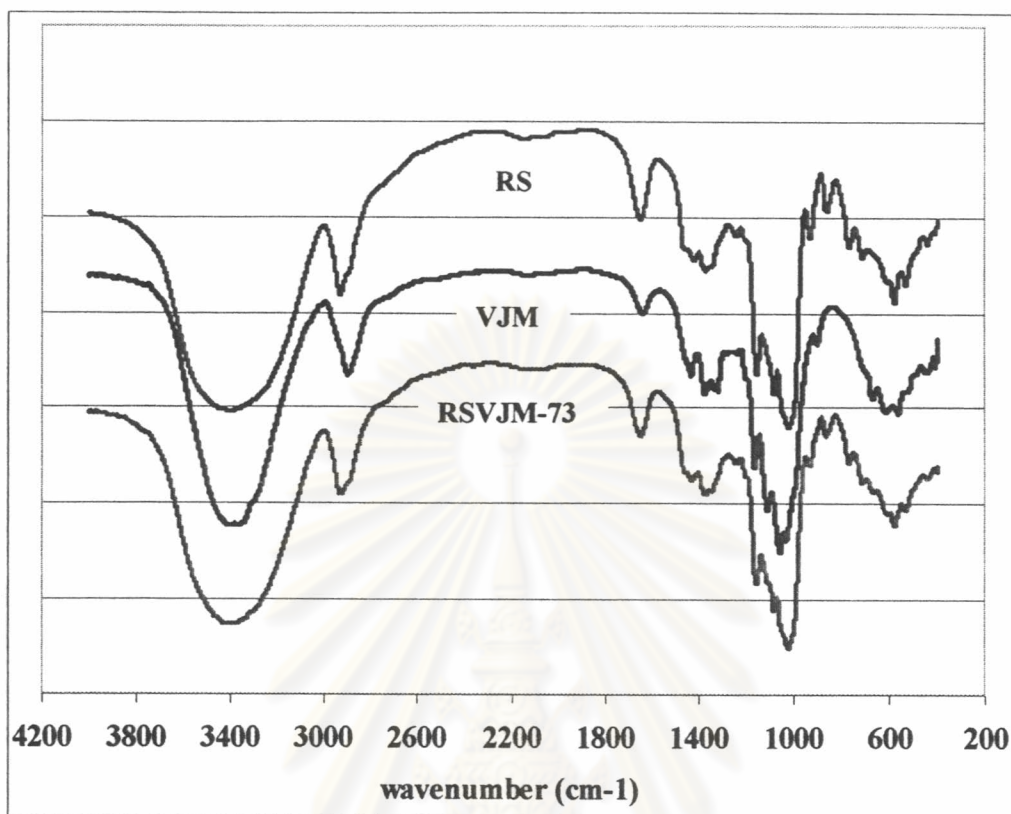


Figure 3-12 IR spectra of RS, VJM, and RSVJM-73.

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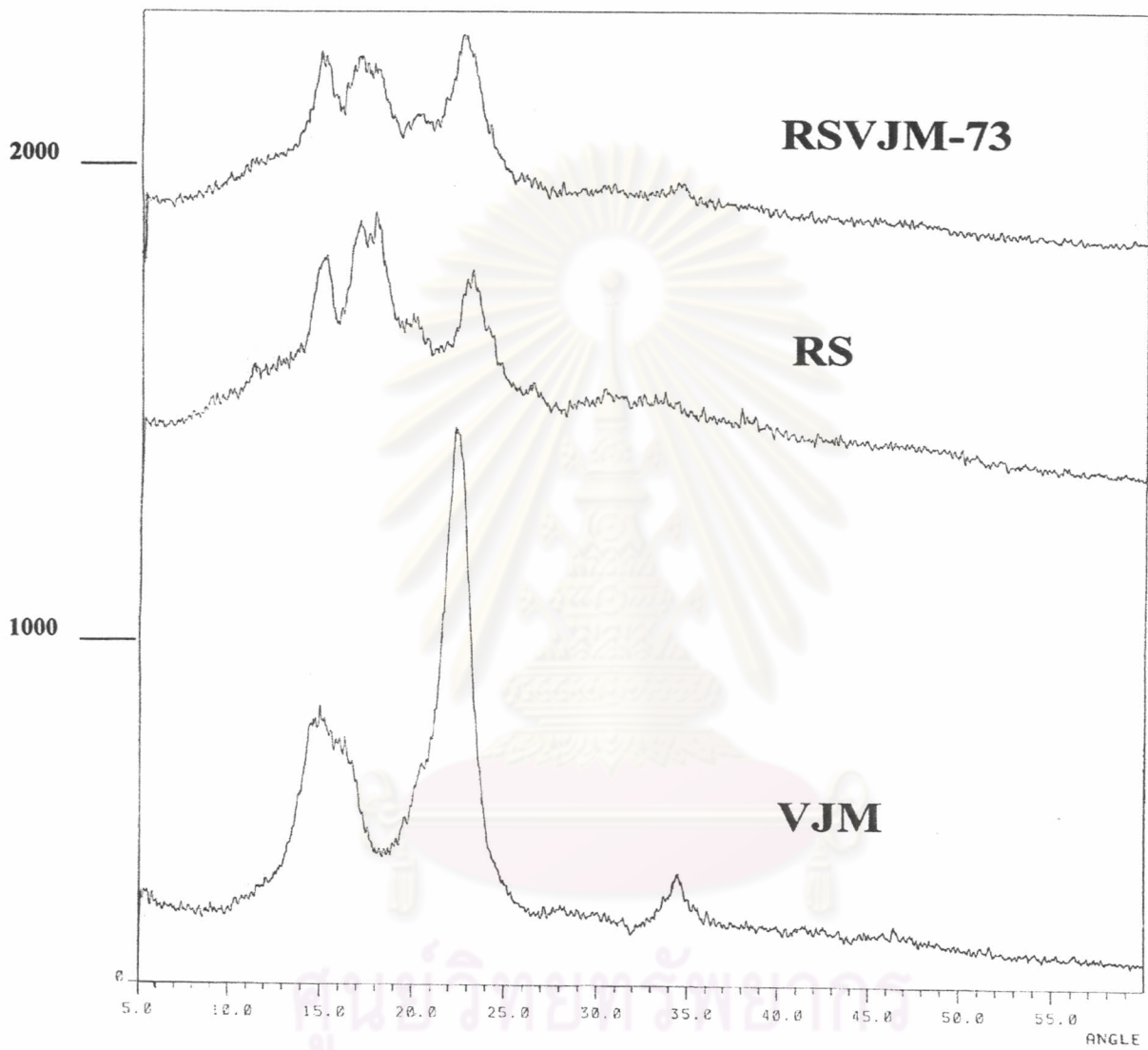


Figure 3-13 X-ray diffractograms of RSVJM-73 , RS, and VJM.

starch in the formulation. Since there was no peak shift or new peak occurred in the IR spectrum of RSVJM-73, indicated no chemical interaction between the starch and cellulose. X-ray pattern of RS is A-type and that of VJM show three peaks at 14.96, 22.40, and 34.52 degree which is similar to Avicel<sup>®</sup> and MCC derived from bagasse in the previous study (Limmatvapirat, 1991). X-ray pattern of RSVJM-73 was similar to the A-type pattern due to the large proportion of starch. No change in the X-ray pattern indicated unchanged of crystallinity in this formulation. However, the peak ratio in RSVJM-73 was different from that of RS, this might be due to the effect of VJM in the formulation.

### Conclusions

Composite particles of starch and MCC were successfully prepared via spray drying technique. The conclusions of this chapter can be summarized as follows:

1. Rice starch had more compressibility properties than rice flour.
2. Increasing MCC in the formulations increased the hardness, reduced %friability and disintegration time of prepared tablets.
3. Spray drying technique produces the spherical form particles of rice starch grains. The higher proportion of MCC in the formulations, the more irregular shape produced, then the flowability of powder is reduced. The formulations prepared with VJM gave the more spherical form of particles than that with VS.
4. In comparing the powder and tableting properties of RSVS-73 and RSVJM-73 with other commercial DC diluents, the selected spray dried formulations gave higher flowability than lactose-based filler; Tablettose<sup>®</sup> and Cellactose<sup>®</sup>; and MCC while lower than that of Eratab<sup>®</sup>. Moreover, the physical properties of the tablets: hardness, %friability, and disintegration time; were superior to lactose-based filler and Eratab<sup>®</sup>.
5. An investigation of IR spectra, X-ray diffractograms and DSC thermograms, spray drying technique appeared to have no effect on the crystallinity or chemical property of starch grain. However, in the formulation of co-dried with MCC which had hydration property, an increase in gelatinization of starch grain might be obtained. And this might be increase tablet strength in addition to the compressibility enhancement of MCC.