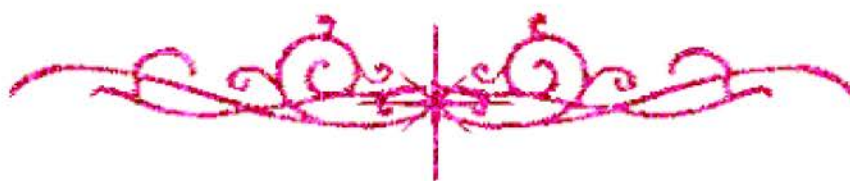


سامية محمد مصطفى



شبكة المعلومات الجامعية

بسم الله الرحمن الرحيم



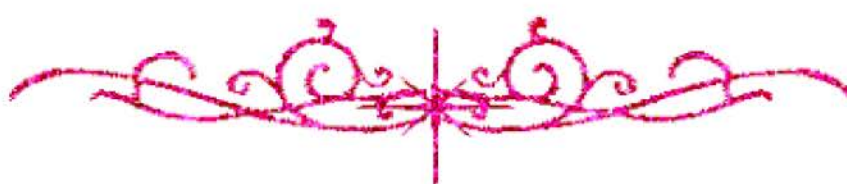
سامية محمد مصطفى



شبكة المعلومات الجامعية



شبكة المعلومات الجامعية التوثيق الالكتروني والميكرو فيلم



سامية محمد مصطفى



شبكة المعلومات الجامعية

جامعة عين شمس

التوثيق الإلكتروني والميكروفيلم

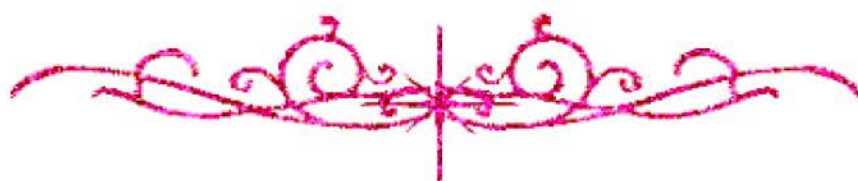
قسم

نقسم بالله العظيم أن المادة التي تم توثيقها وتسجيلها
علي هذه الأقراص المدمجة قد أعدت دون أية تغيرات



يجب أن

تحفظ هذه الأقراص المدمجة بعيدا عن الغبار



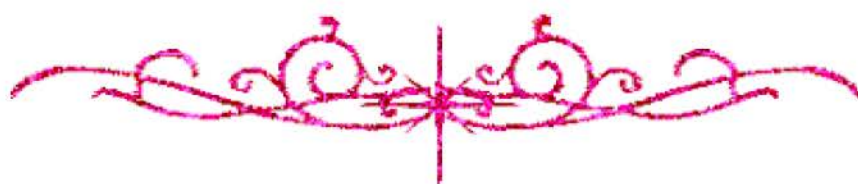
سامية محمد مصطفى



شبكة المعلومات الجامعية



بعض الوثائق الأصلية تالفة



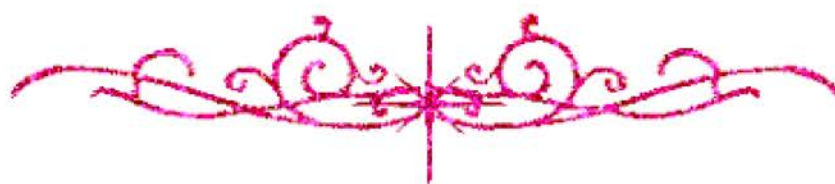
سامية محمد مصطفى



شبكة المعلومات الجامعية



بالرسالة صفحات لم ترد بالأصل



GRANULATION OF FINE POWDER BY FLUIDIZED - BED TECHNIQUE

THESIS

**Submitted in partial fulfillment for the degree of
M. Sc. in Chemical Engineering**

By

Eng. Kamel Mohamed Mahmoud El-Khatib

Under The Supervision of

**Prof. Dr. Hamdy Abd El- Aziz Moustafa
Head of chemical Engineering Department
Faculty of Engineering , Cairo University**

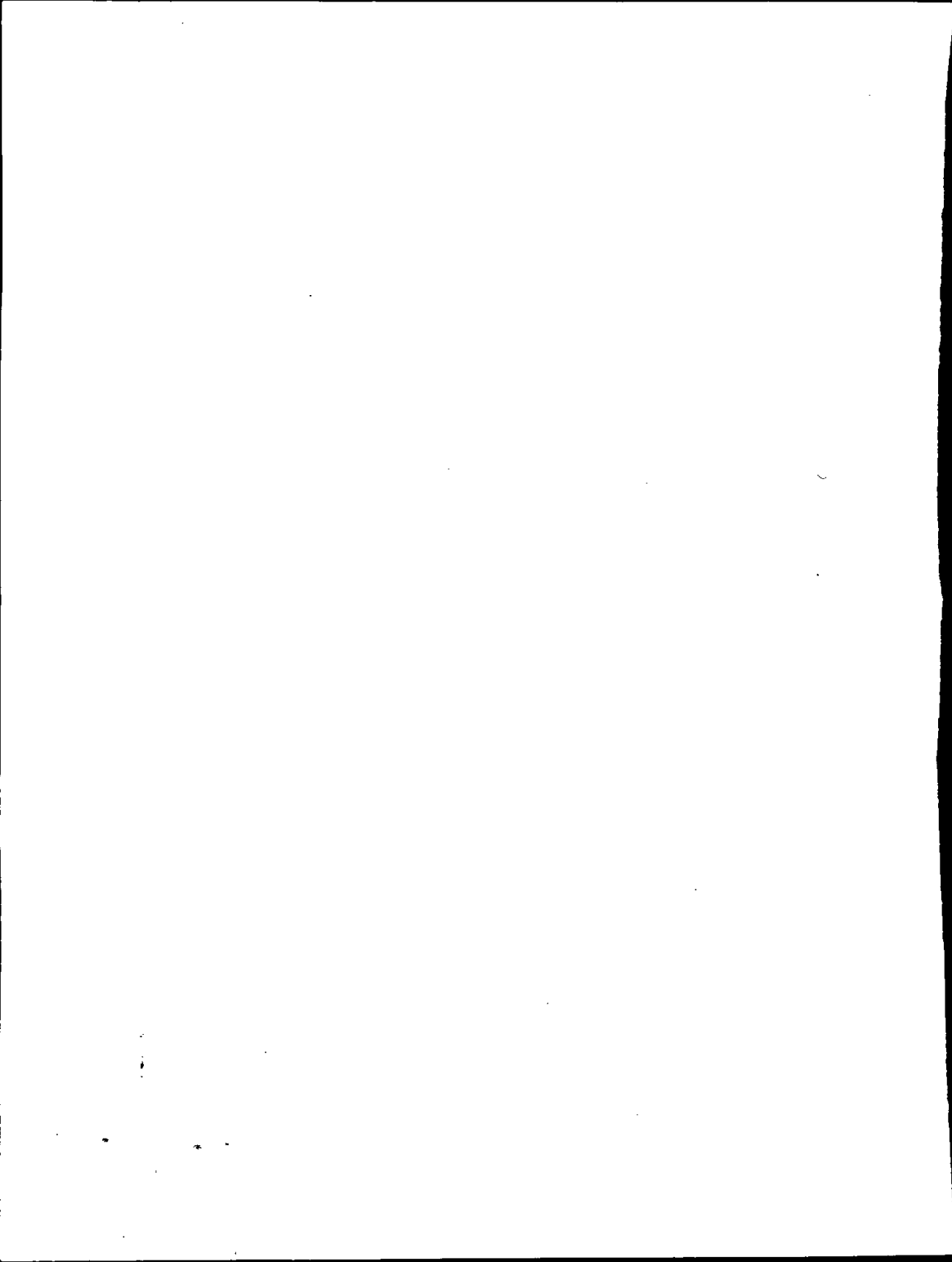
**Prof. Dr. Sohair El- Nawawy
Pilot Plant Laboratory
National Research Center**

**Cairo University
Faculty of Engineering**

1994

B

17-22



ACKNOWLEDGEMENT

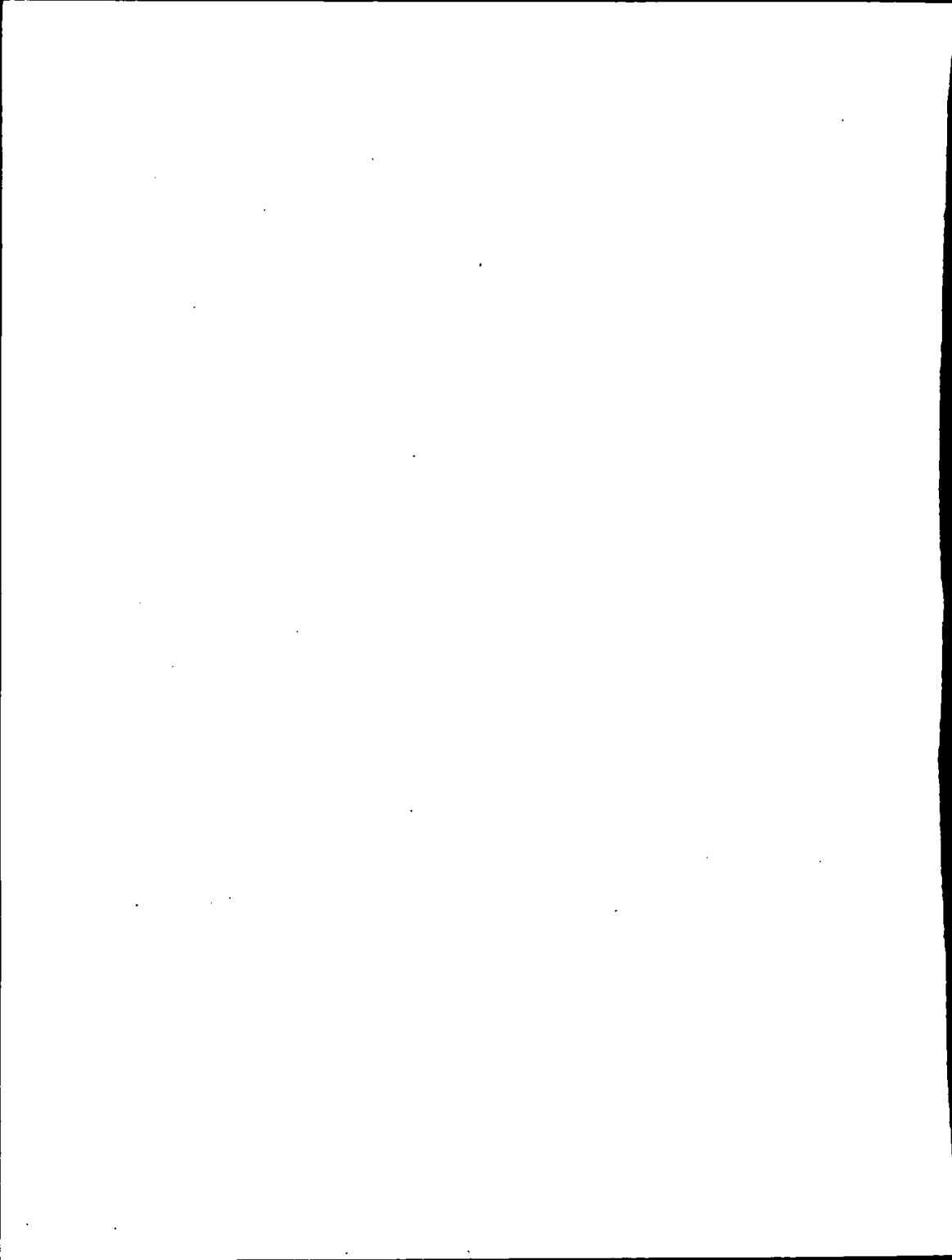
The author wishes to express his sincere appreciation and gratitude to Prof. Dr. H. A. Moustafa, Head of Chemical Engineering Department, Faculty of Engineering, Cairo University, for sponsoring this work, continuous encouragement and helpful discussion.

The author is greatly indebted to Dr. Sohair EL-Nawawy, Associate prof., Pilot Plant Laboratory, National Research Center, for her planning and capable supervision of this work and for offering every possible help and guidance through the whole investigation.

The author thanks Prof. Dr. M. M. El-Halwagi first undersecretary of ministry of scientific research for proposing the point of this research.

The author wishes to thank his COLLEAGUES in The Pilot Plant ,for their sincere help.

Thanks are also due to the National Research Center, for the provision of laboratory facilities.



ABSTRACT

Fluidized bed granulation process has attracted interest in recent years. One of the main advantages of this process over the conventional granulation methods is that different operations such as granulation, mixing, evaporation, and drying are performed in one vessel.

Batch of a mixture (4:1) of lactose and starch which is a common filler material in pharmaceutical industry is used as a raw material in our study.

In this study we investigate the effect of operating parameters on mean particle size, particle size distribution, and physical properties of granules. Operating parameters are superficial gas velocity ranging from 1.5 to 3.5 U_{mf} , bed load ranging from 1 to 1.5 Kg, nozzle height above distributor ranging from 25 to 45 cm, inlet air temperature ranging from 30 to 60 °C. Two types of binder solution are used gelatine and sodium carboxy methyl cellulose with different concentration ranging from 2 % to 8 % w/w for gelatine and from 0.5 % to 1.25 % w/w for sodium carboxy methyl cellulose. Binder solution flow rate ranging from minimum and critical binder solution flow rate (3 to 5 ml/min) , with quantity of binder solution ranging from 110 to 213 ml, and air atomization mass flow rate ranging from 19.75 to 27.2 g/min are used.

In this study it was found that the mean particle size increases with increasing concentration, flow rate, and quantity of binder solution, and decreases with increasing air fluidization

velocity, bed load, and inlet air temperature, but there is no change in mean particle size with the change in atomization air mass flow rate. Type of binder solution has a significant effect on mean particle size and particle size distribution. It was found also that the cumulative weight percent under size decreases with increase of concentration, flow rate, and quantity of binder solution, and with decrease in atomization air mass flow rate. The amount of particles not granulated decreases with increasing of quantity of binder solution, where % < 90 μm reaches to 0.6 % at 4% w/w gelatine solution with 3 ml/min flow rate, 213 ml quantity of binder solution, and 27.2 g/min air nozzle mass flow rate. The cumulative weight percent > 300 μm reach to 51.71 % w/w at the same condition.

The investigation of the physical properties of granule product shows that the granule friability decrease with increasing concentration, flow rate, and quantity of binder solution, and increases with increasing superficial gas velocity, bed load, inlet air temperature, and atomization air mass flow rate. An increase in granule porosity achieved with increasing concentration, and quantity of binder solution, and air nozzle mass flow rate. The granule flowability decreases with increasing granule size.

The optimum binder solution flow rate ranging from 3 to 5 ml/min, nozzle height is 35 cm, and bed height is the height at which there is no channelling and slugging. The maximum viscosity of binder solution to give good atomization of binder is 25 Cp.

CONTENTS

	Page
CHAPTER (1) INTRODUCTION	1
CHAPTER (2) AIM OF WORK	4
CHAPTER (3) THEORETICAL ASPECTS	5
3.1. Fluidization phenomenon	5
3.2. Application of fluidized bed	8
3.3. Granulation technology	9
3.3.1. Objective of granulation process	10
3.3.2. Methods and application of granulation	10
3.3.2.1. Pressure compacting	11
3.3.2.1.1. Confined pressure device	12
3.3.2.1.1.a. Piston or molding presses.....	12
3.3.2.1.1.b. Tableting presses	12
3.3.2.1.1.c. Roll presses	13
3.3.2.1.1.d. Pellet mills	14
3.3.2.1.1.e. Screw extruder	14
3.3.2.2. Tumbling and mixer agglomeration	15
3.3.2.2.1. Tumbling agglomerators	15
3.3.2.2.1.1. Inclined pan or disc agglomerator....	15
3.3.2.2.1.2. A drum agglomerator	16
3.3.2.2.2. Mixer agglomerator	17
3.3.2.2.2.1. Pug mills (Pug, and paddle mixers)....	17
3.3.2.2.2.2. Continuous flow - jet mixing	18
3.3.2.3. Thermal processes	19
3.3.2.3.1. Sintering and heat hardening	19
3.3.2.3.2. Drying and solidification	21

3.3.2.4. Spray method	21
3.3.2.4.1. Spray drying	22
3.3.2.4.2. Prilling	22
3.3.2.4.3. Fluid - and spouted - bed granulator.....	23
3.3.2.4.4. Flash drying.....	24
3.3.2.5. Liquid systems	24
3.3.2.5.1. Wetting with immiscible liquid	24
3.3.2.5.2. Sol - Gel process	25
3.3.2.5.3. Pellet flocculation	26
3.4. Bonding mechanisms in agglomerates	26
3.4.1. Solid bridges	27
3.4.1.1. Adhesion by sintering	27
3.4.1.2. Crystallization and changes of structure ...	28
3.4.2. Adhesion by free mobile liquids	29
3.4.2.1. Liquid bridges between particles	29
3.4.2.2. Bonding mechanisms by a capillary liquid in a completely or partly filled pores of the agglomerates	30
3.4.3. Attraction between solid particles by Van der Waal and electrostatic forces	30
3.4.3.1. Van der Waal adhesion force	30
3.4.3.2. Electrostatic forces	32
3.4.3.2.1. Electrostatic attraction through contact potential	32
3.4.3.2.2. Electrostatic attraction through excess charges	33
CHAPTER(4) REVIEW OF PREVIOUS WORK	35
4.1. Apparatus parameters.....	36

4.1.1. Air distributor.....	36
4.1.2. Shape of granulator body	37
4.1.3. Nozzle height	37
4.2. Process parameters.....	39
4.2.1. Bed load	40
4.2.2. Fluidizing air flow rate	40
4.2.3. Fluidizing air temperature	42
4.2.4. Atomization	43
4.2.4.1. Nozzle type	43
4.2.4.2. Spray angle	44
4.2.4.3. Liquid flow rate	45
4.2.4.4. Atomizing air flow rate	50
4.2.4.5. Atomizing air pressure	51
4.2.4.6. Droplet size	51
4.3. Product parameters	53
4.3.1. Type of binder	53
4.3.2. Quantity of binder solution	54
4.3.3. Binder solvent	55
4.3.4. Concentration of binder solution	56
4.3.5. Temperature of granulating solution	58
4.4. Review on the application of fluidized bed granulator..	58
4.4.1. Pharmaceutical application	58
4.4.2. Steel industry application	59
4.4.3. Glass industry application	61
4.4.4. Fertilizer industry application	61
4.4.5. Water desalination application	63
4.4.6. Metallurgy application	64
4.4.7. Air pollution application	64

CHAPTER (5) EXPERIMENTAL SET-UP AND PROCEDURE	66
5.1. Base material	66
5.2. Binder solution	66
5.2.1. Preparation of binder solution	66
5.3. Experimental apparatus	68
5.3.1. Fluidized bed chamber	68
5.3.2. Air system	71
5.3.2.1. Air supply line	71
5.3.2.2. Air heater	71
5.3.2.3. Air distributor	73
5.3.2.4. Cyclone (Gas-Solid separation system) ..	73
5.3.3. Water system	75
5.3.3.1. Atomizer (Pneumatic binary nozzle)	75
5.3.3.2. Water pump	77
5.3.3.3. Humidity determination	77
5.4. Experimental procedure	77
5.5. Analytical techniques.....	79
5.5.1. Determination of mean particle size and particle size distribution	79
5.5.2. Granule friability	79
5.5.3. Bulk density	80
5.5.4. Granule density	80
5.5.5. Granule porosity	80
5.5.6. Granule flowability	81
CHAPTER (6) RESULTS AND DISCUSSION	82
6.1. Introduction	82
6.2. The relation between pressure drop through the bed and superficial gas velocity	83