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(54) Title: FLOCCULATING AGENT FOR WASTE WATER TREATMENT AND METHOD OF USING THEREOF

(57) Abstract: A flocculating agent for waste water treatment, comprising, by weight, 2.52% to 12.52% Na<sub>2</sub>O, 43.25% to 53.25% SiO<sub>2</sub>, 16.52% to 26.52% Al<sub>2</sub>O<sub>3</sub>, 1 % to 6.06% K<sub>2</sub>O, 1 % to 10.22% CaO, 1 % to 8.25% Fe, 1.12% to 11.12% chitosan, and 2.06% to 12.06% ceramic powder, wherein the particle size of each said components is approximately 50 pm. Preferably, the flocculating agent comprises, by weight, 7.52% Na<sub>2</sub>O, 48.25% SiO<sub>2</sub>, 21.52% Al<sub>2</sub>O<sub>3</sub>, 1.06% K<sub>2</sub>O, 5.22% CaO, 3.25% Fe, 6.12% chitosan, and 7.06% ceramic powder. The amount of flocculating agent added into waste water to satisfactorily agglomerate and flocculate wastes and other contaminants in the forms of suspended solids and colloidal particles is based on types of waste water.



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## **FLOCCULATING AGENT FOR WASTE WATER TREATMENT AND METHOD OF USING THEREOF**

### **TECHNICAL FIELD OF THE INVENTION**

5 This invention is related to a flocculating agent for waste water treatment and method of using thereof, and more particularly, to a flocculating agent for agglomerating and flocculating wastes and other contaminants from waste water, and the method of using thereof.

### **BACKGROUND OF THE INVENTION**

Nowadays, contamination of water in seas, rivers, ponds, and lakes is increasing due to a variety of reasons, such as shortcoming of sewage system, oil spill, marine dumping, eutrophication, etc. Aside from that, one of the major contributors in water contamination is industrial waste disposal in  
15 highly concentrated industrial areas, especially those areas that are closely situated to significant water sources. Many industrial facilities use freshwater to carry away waste from the plant and into rivers, lakes and oceans. Moreover, due to the extensive water use and discharges of insufficiency treated or even not treated waste water by the industries, clean water sources  
20 and industrial water sources are depleting. A further main cause of water pollution is the ever increasing city population, which has caused domestic waste water to be increased. Therefore, supplying industries and homes with fresh water has become increasingly more difficult.

25 Therefore, treatment of waste water in order to reuse the water is vital, whether it is for human consumption, industrial uses, or for the sake of protecting the environment. Various methods of water treatment has been known and used for the aforesaid purposes. For one instance, water is treated with chlorine-releasing agents for human consumption purpose. However,  
30 water treated by chlorine-releasing agents has a number of drawbacks, such as unpleasant smell and taste. In addition, when phenols are present in chlorinated water, the phenols will react with the chlorine to generate trichlorophenol, which increase the severity of the unpleasant taste and smell

of the chlorinated water. Another drawback of chlorine-treated water is that the chlorine-releasing agents do not agglomerate and/or flocculate colloidal particles and suspended solids in the water as it only works to kill off the bacteria present in the water.

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Another known method for treating water is the addition of flocculating agent, such as aluminium sulphate, inorganic low-molecular flocculating agents, inorganic polymer flocculating agents, organic polymer flocculating agents, gelatin, and silica gel, into waste water to agglomerate and flocculate waste  
10 from waste water. This technology has been widely used in treatment of sewage waste water, drinking water, industrial waste water, etc. Nonetheless, these well-known flocculating agents have limitations in their uses.

Even though aluminium sulphate is the most commonly used flocculant  
15 because of its relatively low cost production, it is not a very effective flocculating agent. For examples, the agglomeration and flocculation speed is very low, and the size of formed flocs is small. Further, use of aluminium sulphate must be accompanied by high temperature in order to perform adequate aggregation. Furthermore, aluminium sulphate causes the treated  
20 water to be highly acidic, which in turns, requires input of large amount of basic materials for neutralization purpose.

Ferric chloride, which belongs to the group of inorganic low-molecular flocculating agents, however, shows stronger agglomeration and flocculation  
25 properties as formation of flocs is larger and it is sufficiently efficient in agglomerating and flocculating wastes as well as heavy metals. Nevertheless, ferric chloride solution is highly corrosive and has low stability. Apart from that, the use of ferric chloride in treating waste water must be accompanied by slaked lime, which causes large production of sludge. Baking such sludge in  
30 turn causes further environmental pollution, which is not sensible.

Therefore, it is an aim of this present invention to address the aforesaid technical disadvantages by introducing a novel and inventive flocculating

agent, which is used in treating waste water resulting from domestic, commercial, and industrial discharges. The flocculating agent works by agglomerating and flocculating wastes and other contaminants in the forms of suspended solids and colloidal particles from the waste water.

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## **SUMMARY OF THE PRESENT INVENTION**

The present invention relates to a novel and inventive flocculating agent for treating waste water caused by domestic, commercial, and industrial discharges. The flocculating agent acts by agglomerating and flocculating  
10 wastes and other contaminants in the forms of suspended solids and colloidal particles into flocs or flakes. The flocs may then float to the top of the liquid, or settle to the bottom of the liquid, which are then readily filtered from the liquid.

In an embodiment of this present invention, the flocculating agent comprises,  
15 by weight, 2.52% to 12.52%  $\text{Na}_2\text{O}$ , 43.25% to 53.25%  $\text{SiO}_2$ , 16.52% to 26.52%  $\text{Al}_2\text{O}_3$ , 1% to 6.06%  $\text{K}_2\text{O}$ , 1% to 10.22%  $\text{CaO}$ , 1% to 8.25%  $\text{Fe}$ , 1.12% to 11.12% chitosan, and 2.06% to 12.06% ceramic powder.

In a preferred embodiment of this present invention, the flocculating agent  
20 comprises, by weight, 7.52%  $\text{Na}_2\text{O}$ , 48.25%  $\text{SiO}_2$ , 21.52%  $\text{Al}_2\text{O}_3$ , 1.06%  $\text{K}_2\text{O}$ , 5.22%  $\text{CaO}$ , 3.25%  $\text{Fe}$ , 6.12% chitosan, and 7.06% ceramic powder.

In another embodiment of this invention, the particle size of each aforesaid components present in the flocculating agent is approximately 50  $\mu\text{m}$ .

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In a further embodiment of this invention, the flocculating agent can be used to treat waste water resulting from domestic, commercial, and industrial discharges, such as water soluble coating waste water, water soluble mechanical waste water, factory waste water, construction site waste water,  
30 concrete waste water, iron(III) oxide waste water, chromium waste, arsenic waste water, sludge, other contaminated liquids, and the like.

In yet another embodiment of this invention, the flocculating agent is further employed for decolouration of dye waste water, print waste water, pigment waste water, and the like.

- 5 In still another embodiment of this invention, the amount of the flocculating agent that is required to satisfactorily treat the waste water is 100 mg/L to 4000 mg/L.

#### **BRIEF DESCRIPTION OF THE DRAWINGS**

- 10 Figure 1 illustrates the effect of the flocculating agent on water soluble coating waste water according to the present invention;

Figure 2 illustrates the effect of the flocculating agent on water soluble mechanical waste water according to the present invention;

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Figure 3 illustrates the effect of the flocculating agent on factory waste water according to the present invention;

- 20 Figure 4 illustrates the effect of the flocculating agent on construction site waste water according to the present invention;

Figure 5 illustrates the effect of the flocculating agent on concrete waste water according to the present invention;

- 25 Figure 6 illustrates the effect of the flocculating agent on iron(III) oxide waste water according to the present invention;

Figure 7 illustrates the effect of the flocculating agent on chromium waste water according to the present invention; and

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Figure 8 illustrates the effect of the flocculating agent on arsenic waste water according to the present invention.

**DETAILED DESCRIPTION OF THE PRESENT INVENTION**

The above mentioned and other features and objects of this invention will become more apparent and better understood by reference to the following detailed description. It should be understood that the detailed description made known below is not intended to be exhaustive or limit the invention to the precise form disclosed as the invention may assume various alternative forms. On the contrary, the detailed description covers all the relevant modifications and alterations made to the present invention, unless the claims expressly state otherwise.

In accordance with the present invention, there is disclosed a novel and inventive flocculating agent that is employed to treat waste water by agglomeration and flocculation of wastes and other contaminants that are in the forms of suspended solids and colloidal particles. The agglomerated and flocculated wastes, which are also known as either flocs or flakes, may then float to the top of the liquid or settle to the bottom of the liquid, readily and easily to be filtered from the liquid.

The flocculating agent comprises, by weight, 2.52% to 12.52%  $\text{Na}_2\text{O}$ , 43.25% to 53.25%  $\text{SiO}_2$ , 16.52% to 26.52%  $\text{Al}_2\text{O}_3$ , 1% to 6.06%  $\text{K}_2\text{O}$ , 1% to 10.22%  $\text{CaO}$ , 1% to 8.25%  $\text{Fe}$ , 1.12% to 11.12% chitosan, and 2.06% to 12.06% ceramic powder. Preferably, the flocculating agent comprises, by weight, 7.52%  $\text{Na}_2\text{O}$ , 48.25%  $\text{SiO}_2$ , 21.52%  $\text{Al}_2\text{O}_3$ , 1.06%  $\text{K}_2\text{O}$ , 5.22%  $\text{CaO}$ , 3.25%  $\text{Fe}$ , 6.12% chitosan, and 7.06% ceramic powder. The particle size of each aforesaid components is approximately 50  $\mu\text{m}$ .

The flocculating agent is highly effective in treating a variety of waste water, resulting from domestic, commercial, and industrial discharges, such as waste soluble coating waste water, water soluble mechanical waste water, factory waste water, construction site waste water, concrete waste water, iron(III) oxide waste water, chromium waste water, arsenic waste water, sludge, other contaminated liquids, and the like. The flocculating agent is also highly

effective in decolouration of dye waste water, print waste water, pigment waste water, and the like.

The amount of the flocculating agent necessary for effective treatment of waste water is actually based on types of waste water. Refer to Table 1 for non-exhaustive examples of the amounts of flocculating agent needed for treating different species of waste water. In general, 100 mg/L to 4000 mg/L of the flocculating agent is added into waste water to produce satisfactorily agglomeration and flocculation results. Upon addition of the flocculating agent, the waste water is vigorously stirred for approximately 10 seconds and left to stand for an additional 10 seconds to visualize the formation of flocs. The waste water temperature and pH do not affect the performance of the flocculating agent. Therefore, pre-treatment and post-treatment of the waste water with heat, and basic or acidic chemicals are not necessary, resulting in further cost reduction. Figures 1 to 8 illustrate the conditions of different types of waste water before and after the addition of the flocculating agent.

Table 1: Amounts of flocculating agent needed to treat different types of waste water

Types of waste water	Amount (mg/L)
Water soluble coating waste water	4000
Water soluble mechanical waste water	4000
Factory waste water	150
Construction site waste water	150
Concrete waste water	200
Iron(III) oxide waste water	200
Chromium waste water	200
Lake waste water	20 – 50
Reservoir waste water	20 – 50
Fish farm waste water	10 – 30
Arsenic waste water	100

Industrial waste water treated with the flocculating agent in accordance with the present invention is tested for harmful and toxic components with various detection methods, such as chromatography, spectrophotometer, and JIS K 0102. The test results are shown in Table 2. According to the test results, harmful and toxic components, for instances alkyl mercury, mercury compound, cadmium compound, lead, phosphorus, chromium, arsenic, hydrogen cyanide, phenyl chloride, copper, zinc, fluorine, and the like, are not present or detected in the treated industrial waste water.

Table 2: Detection of harmful and toxic material in industrial waste water treated with flocculating agent

Harmful components	Result	Detection method
Alkyl mercury	Not detected (<0.0005)	Chromatography
Mercury compound	Not detected (<0.0005)	AA spectrophotometer
Cadmium compound	Not detected (<0.002)	JIS K 0102 55.2. AA
Lead	Not detected (<0.05)	JIS K 0102 54.2. AA
Phosphorus	Not detected (<0.01)	Gas chromatography
Chromium	Not detected (<0.05)	JIS K 0102 65.2.1. ICP
Arsenic	Not detected (<0.09)	JIS K 0102 61.1. ICP
Hydrogen cyanide	Not detected (<0.01)	JIS K 0102 38.1.2. & 38.3. AA
Phenyl chloride	Not detected (<0.0005)	JIS K 0093 AA
Copper	Not detected (<0.10)	JIS K 0102 52.2. ICP
Zinc	Not detected (<0.50)	JIS K 0102 53.2. ICP
Fluorine	Not detected (<1.0)	JIS K 0102 34.1. ICP

Waste water from Petronas Melacca refining plant is collected and treated with flocculating agent in accordance with the present invention. The treated sample is analyzed for its amount of contaminants with methods that are published in APHA 19<sup>th</sup> Edition 1995. The results are tabulated and shown below:



Table 3: Waste water treated with the flocculating agent according to the present invention

Test	Waste water treated with flocculating agent	Standard B specification*	Method
pH value	6.44	5.5 – 9.0	APHA 4500-H B
BOD (5 days at 20 °C)	29 mg/L	50 mg/L	APHA 5210 B & APHA 4500 O C
COD	130 mg/L	100 mg/L	APHA 5220 B
Suspended solids	12 mg/L	100 mg/L	APHA 2940 D
Oil and grease	NDLT 2.5 mg/L	10 mg/L	APHA 5520 B
Free chloride	0.31 mg/L	2.0 mg/L	APHA 4500-Cl G
Phenol	NDLT 0.1 mg/L	1.0 mg/L	APHA 5530-D
Sulphide	NDLT 0.2 mg/L	0.5 mg/L	APHA 4500-S <sup>2-</sup> E
Nitrogen (ammonia)	0.9 mg/L	NA mg/L	APHA 4500-NH <sub>2</sub> F

NDLT denotes as None Detected Less Than

NA denotes as not applicable to the stated specification

- 5 \* Malaysian Environment Quality Act 1974, Environment Quality (Sewage & Industrial Effluents) Regulations 1979

Referring to the above-identified results, the contaminant levels of suspended solids, oil and grease are dramatically reduced. Furthermore, the biological oxygen demand (BOD) and chemical oxygen demand (COD) levels of waste water treated with the flocculating agent is significantly lower than standard B specification as stated in Malaysian Environment Quality Act 1974, and

Environment Quality (Sewage & Industrial Effluents) Regulations 1979. BOD is a standard chemical procedure for determining the uptake rate of dissolved oxygen in water by biological organisms residing in said water, whereas COD is a test method to measure indirectly the amount of organic compounds in water. Both the BOD and COD are commonly used in the measurement and indication of water quality. Apart from that, the amounts of free chlorine, phenol, sulphide, and ammonia are significantly reduced as well.

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**I/WE CLAIM**

1. A flocculating agent for treatment of waste water, comprising, by weight,

2.52% to 12.52%  $\text{Na}_2\text{O}$ ;

5 43.25% to 53.25%  $\text{SiO}_2$ ;

16.52% to 26.52%  $\text{Al}_2\text{O}_3$ ;

1% to 6.06%  $\text{K}_2\text{O}$ ;

1% to 10.22%  $\text{CaO}$ ;

1% to 8.25%  $\text{Fe}$ ;

10 1.12% to 11.12% chitosan; and

2.06% to 12.06% ceramic powder;

wherein the particle size of each said components is approximately 50  $\mu\text{m}$ .

15 2. A flocculating agent in accordance with claim 1, wherein the flocculating agent preferably comprises, by weight, 7.52%  $\text{Na}_2\text{O}$ ; 48.25%  $\text{SiO}_2$ ; 21.52%  $\text{Al}_2\text{O}_3$ ; 1.06%  $\text{K}_2\text{O}$ ; 5.22%  $\text{CaO}$ ; 3.25%  $\text{Fe}$ ; 6.12% chitosan; and 7.06% ceramic powder.

20 3. A flocculating agent in accordance with claim 1, wherein the waste water resulting from domestic, commercial, and industrial discharges is but not limited to water soluble coating waste water, water soluble mechanical waster water, factory waste water, construction site waste water, concrete waste water, iron(III) oxide waste water, chromium waste water, arsenic waste  
25 water, sludge, other contaminated liquids, or the like.

4. A flocculating agent in accordance with claim 1, wherein the flocculating agent is further employed for decolouration of dye waste water, print waste water, pigment waste water, and the like.

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5. A method of using a flocculating agent for treatment of waste water, comprising by weight, 2.52% to 12.52%  $\text{Na}_2\text{O}$ , 43.25% to 53.25%  $\text{SiO}_2$ , 16.52% to 26.52%  $\text{Al}_2\text{O}_3$ , 1% to 6.06%  $\text{K}_2\text{O}$ , 1% to 10.22%  $\text{CaO}$ , 1% to 8.25%

Fe, 1.12% to 11.12% chitosan, and 2.06% to 12.06% ceramic powder, wherein the particle size of each said components is approximately 50  $\mu\text{m}$ , is by adding 100 mg/L to 4000 mg/L of said flocculating agent into said waste water, sludge, and/or contaminated liquids.

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6. A method in accordance with claim 5, wherein the flocculating agent preferably comprises, by weight, 7.52%  $\text{Na}_2\text{O}$ ; 48.25%  $\text{SiO}_2$ ; 21.52%  $\text{Al}_2\text{O}_3$ ; 1.06%  $\text{K}_2\text{O}$ ; 5.22%  $\text{CaO}$ ; 3.25%  $\text{Fe}$ ; 6.12% chitosan; and 7.06% ceramic powder.

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7. A method in accordance with claim 5, wherein the waste water resulting from domestic, commercial, and industrial discharges is but not limited to water soluble coating waste water, water soluble mechanical waste water, factory waste water, construction site waste water, concrete waste water, iron(III) oxide waste water, chromium waste water, arsenic waste water, sludge, other contaminated liquids, or the like.

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8. A method in accordance with claim 5, wherein the flocculating agent is further employed for decolouration of dye waste water, print waste water, pigment waste water, and the like.

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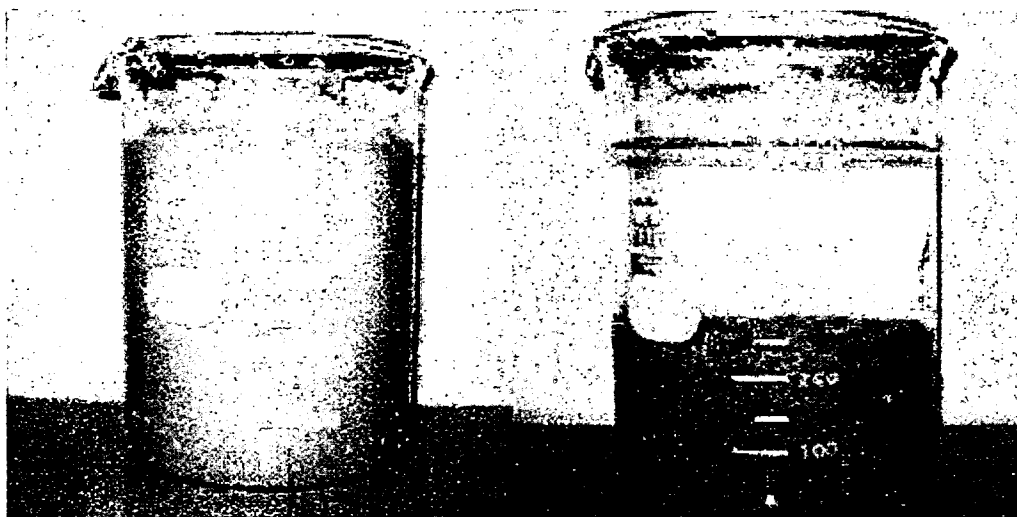


Figure 1

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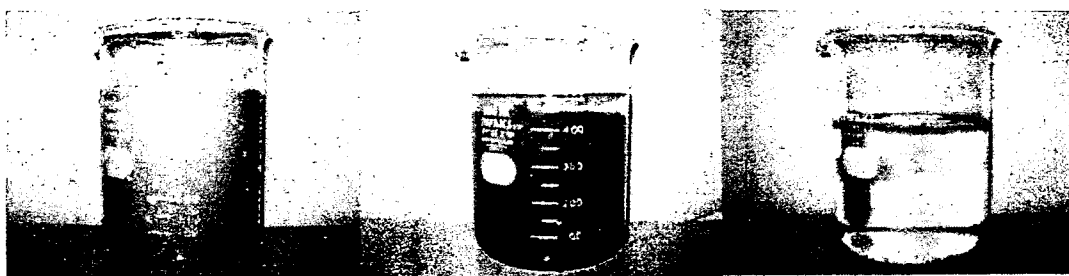
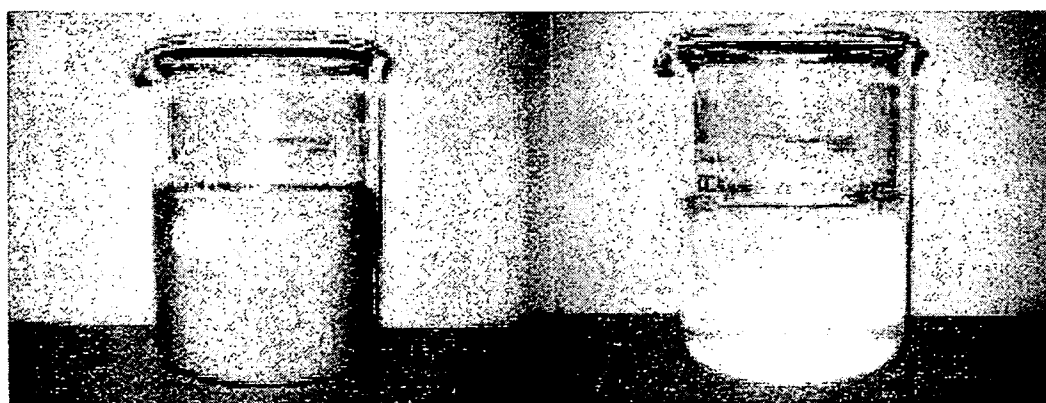


Figure 2

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**Figure 3**

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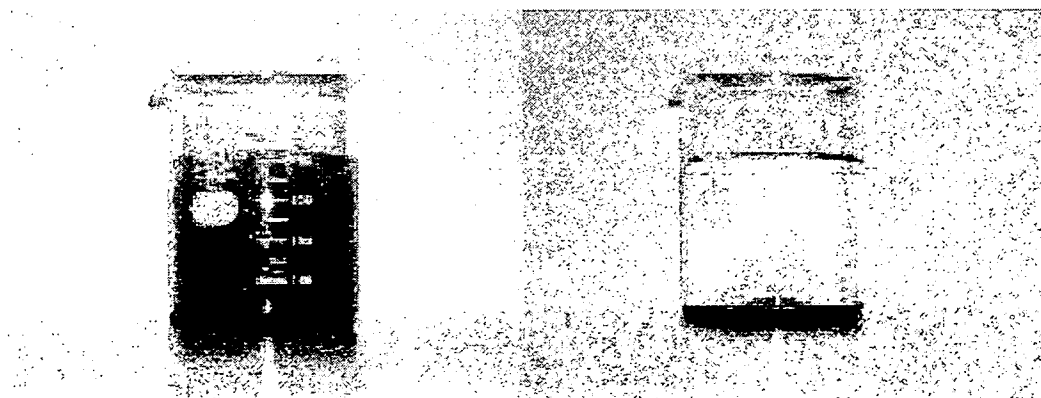


Figure 4



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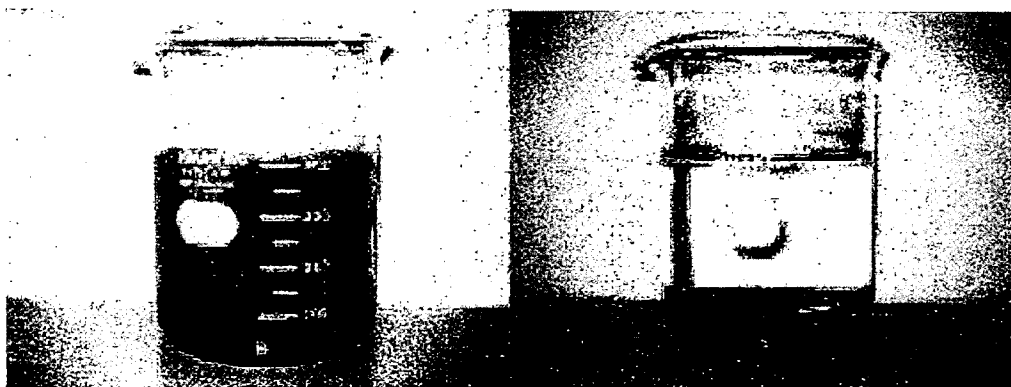


Figure 5

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**Figure 6**

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Figure 7

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Figure 8

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/MY2011/000118

## A. CLASSIFICATION OF SUBJECT MATTER

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C02F 1/52 (2006.01)

C08K 3/10 (2006.01)

C02F 1/56 (2006.01)

C08K 3/20 (2006.01)

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI, EPODOC, NPL: IPC marks - /IC/EC C02F1/52/LOW, C02F1/68, C02F11/14, B01D21/01, C08K3/10, C08K3/20 & keywords: flocculation, coagulation, chitosan, silica, ceramic, sodium oxide, silicon dioxide, silica, aluminium oxide, alumina, potassium oxide, calcium oxide, iron and similar terms; Esp@cenet, Google Patents, Science Direct, Google Scholar (including NPL) & STN using similar keywords.

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4566986 A (WALDMANN) 28 January 1986 Abstract; col. 1, lines 6-8; col. 3, lines 57-62; col. 7, line 13-col. 8, line 41; Example X; claims 1, 5-11	
A	GB 2364047 A (THE PROCTOR & GAMBLE COMPANY) 16 January 2002 Abstract; page 4, lines 14-19; page 5, lines 16-27, page 12, lines 15-27, page 13, lines 27-30, page 14, lines 24-28; Example 1; claims 2, 7	
A	US5393435 A (DEANS et al.) 28 February 1995 Abstract; col. 1, lines 39-62; col. 2, lines 38-47; claims 1-6	



Further documents are listed in the continuation of Box C



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* Special categories of cited documents:	
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Date of the actual completion of the international search

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Date of mailing of the international search report

17.08.2011

Name and mailing address of the ISA/AU

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## INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

**PCT/MY2011/000118**

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report				Patent Family Member			
US	4566986	CA	1259773				
GB	2364047	AR	028760	AU	68659/01	BR	0111953
		CN	1438972	EC	SP024407	EP	1294644
		GB	2364048	GT	200100128	HK	1059922
		JP	2004501750	MA	25823	MX	PA03000081
		PA	8521301	US	2004026657	US	6827874
		WO	0200557	ZA	200209661		
US	5393435	NONE					
Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.							
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