A LABORATORY STUDY OF FLUIDISED DRUM GRANULATION

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ABSTRACT

Performance study of laboratory scale Fluidised Drum Granulation (FDG) has been carried out using glass beads as model particles and melted wax as binder. The equipment was running well to coat the particle. The particle growth was simple, agglomerating or layering. Several process variables such as particle size, binder spray rate and amount of binder added, and drum speed were used to determine the unwanted agglomerate formation. The mass fraction of agglomerate formation increased sharply begin from initial particle size 3 mm to smaller. The transition size is 4 mm, there was virtually no agglomerate formation. Agglomerate formation also increased with increasing binder spray rate, and varies little with drum speed and total amount of binder added.

INTISARI

Percobaan granulasi pada fluidisasi drum pada skala laboratorium telah dilakukan dengan bahan partikel bola gelas dan lilin cair sebagai perekat. Peralatan yang dipakai berjalan dengan baik. Dengan menggunakan partikel bola gelas dan perekat lilin cair tersebut, hanya ada dua pilihan pertumbuhan partikel yaitu, aglomerasi atau pelapisan. Beberapa besaran proses dicoba untuk mengetahui fraksi masa aglomerat yang terbentuk, seperti, ukuran partikel, kecepatan penyemprotan perekat dan jumlah total perekat dan kecepatan putar dari drumnya. Pengaruh ukuran partikel dari 3 mm turun ke 1,5 mm memberi jumlah agglomerat yang terbentuk bertambah secara menyolok. Pembentukan agglomerat juga bertambah dengan bertambahnya kecepatan penyemprotan perekat, dan sedikit bertambah dengan berkurangnya kecepatan putar dari drumnya dan juga jumlah total perekat yang ditambahkan.

INTRODUCTION

The development of granulation devices is becoming more and more important in the pharmaceutical, chemical, ceramic, cement, powdered metal, non-ferrous metal, iron and steel industries. Granulation converts powders, filter cakes, pastes and similar materials into free flowing, easy to handle, granules with may also show better dispersibility and wet-ability than the powdered materials. Granulation also gives better surface conditions for drying, firing and better flow properties.

There are many granulation devices and methods available depending on the physical properties and type of the materials to be granulated. These include inclined pans, pugmills or blungers, rotary drum and fluidised and spouted beds granulators. Each of these types of granulator have been shown to have advantages in some applications. There are several detailed reviews of granulator types [1].

The traditional process particles tumble in the pan and growth occurs [2]. For coating pans or drums, the adhesive or coating materials are added sequentially during particle tumbling. In this system, generally growth rate is critically dependent on moisture content and particle size, and the particle growth mechanism is complex. Because this is a complex process, it is difficult to control which mechanism is dominant, and the product size distribution is difficult to predict resulted broader size distribution.

The key issue in the fertilizer granulation process is high recycle ratios, up 1 : 6 [3]. This is due to the broad product size distribution leaving from the granulator. To improve that ratio, the main goal is to get a narrower product size distribution which can be achieved if layering mechanism dominates. To get layer growth, new feed is in the form of melt or solution to slowly build layers [4].

Fluid beds coater are using the feed in the form of melt or solution. Feed solutions such as ammonium sulfate [5], sodium chloride [6], and sulphur [7] have been used. In thermal granulation processes, the feed is a melt. The melt is preheated to a temperature above its melting point and the hot liquid sticking to the particle is cooled down and solidified by the fluid gas.

The main particle growth mechanisms are agglomeration or layering. If the liquid bridge is strong enough to hold the particles together, agglomeration takes place. If break-up forces are stronger, layering is the dominant process. By manipulation of process and operation conditions one of these mechanisms will dominate. The effect of process variables on the particle growth in fluidised bed granulation was observed by Smith and Nienow [8].

Increasing particle feed size and gas fluidisation velocity, and reducing binder viscosity lead the changing of growth mechanism from agglomeration to layering. An excellent review of fluidised process has been presented by Nienow [9].

The continuous FDG process (Figure 1) is similar to an earlier falling curtain granulator reported by Shirley [10], for urea granulation. He improved the drum granulator by installing a collecting pan and heat removal system.

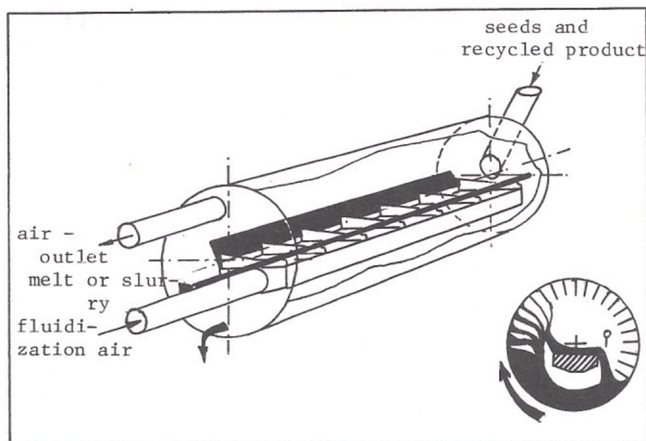


Figure 1. Schematic of a continuous fluidised drum granulator [11].

The FDG process was first developed in 1986 by Kaltenbach-Thuring, France [11]. There are three major advantages of FDG process; that are simplicity, quality of granules, and the possibility of retrofitting the process into existing units [11]. A full scale FDG was installed by Incitec in Newcastle, NSW-Australia, to produce granular ammonium nitrate. The major criterion in the process selection was the minimisation of the capital cost, limitation of effluent discharges, and minimisation of operator surveillance. The recycle ratio is low, and air pollutant abatement good [12]. Despite its industrial success, fundamental studies of FDG operation have not been reported in the literature. The factors that affect performance especially formation of unwanted agglomerates have not been identified.

The purpose of this study is to design and run a laboratory scale of FDG using model materials in order to understand its performance and characteristic in conjunction to the formation of unwanted agglomerates.

MATERIALS AND EQUIPMENT

MATERIALS

The materials used for this study were glass beads as the particles and wax as binder. The physical properties of these materials are summarised as follow:

Seven different sizes of soda glass beads (1.5, 2, 3, 4, 5, 6, and 8 mm in diameter) are used in this work. All soda glass beads are rigid and non-porous. The beads were supplied by Australian Scientific Pty.Ltd. The particle diameter was measured to ± 0.01 mm using a digimatic Caliper (Mitutoyo Corporation, Japan). For each size of bead, 50 particles were sampled and measured to give the data in Table 1.

Table 1. Glass bead properties

Average size (mm)	Standard deviation (mm)	Average weight/ particle (g)	Standard deviation (g)	Number of particles/kg
1.475	0.044	0.00437	0.00018	228520
1.899	0.046	0.00983	0.00056	101681
2.906	0.031	0.03510	0.00097	28531
3.959	0.052	0.08290	0.00204	12065
4.832	0.052	0.15540	0.00227	6436
6.126	0.070	0.31190	0.00265	3206
8.040	0.072	0.70310	0.01618	1422

The true particle density was calculated by measuring the average of particle diameter and mass, assuming the beads to be a perfect spheres. The true particle density is 2.64 ± 0.1 g/cm^3 for all sizes.

Paraffin wax (melting point in a range of 56 - 61°C) is used as a binder to coat the particles. Molten wax is used as a binder by melting the feed wax at temperature to 150°C - 160°C. The advantage of the wax binder is that a solvent is not required. A solid coating is achieved simply by cooling and solidifying the wax. As there is no solvent evaporation, the coating mass balance is simplified.

EQUIPMENT

The equipment used in this work is presented schematically in Figure 2. Laboratory experiments were carried out in a stainless steel drum of diameter 0.3 m and length 0.2 m. The drum contains six lifters each 4 cm high. Both end sides are covered by transparent perspex covers, so one can observe particle motion in the drum. On one side, the cover is removable to load and unload the particles. There is also a hole in this cover 160 mm in diameter for the air outlet and also for the air table and nozzle installation. This hole is loosely covered during operation to ensure no loss of particles bouncing off the air table. The drum is mounted on four roller wheels (two on either side) controlled by a variable speed drive (Model TL 362, mfg. no. 33722, Mangrovite, Australia) which allows the drum speed to be varied from 0 - 80 rpm.

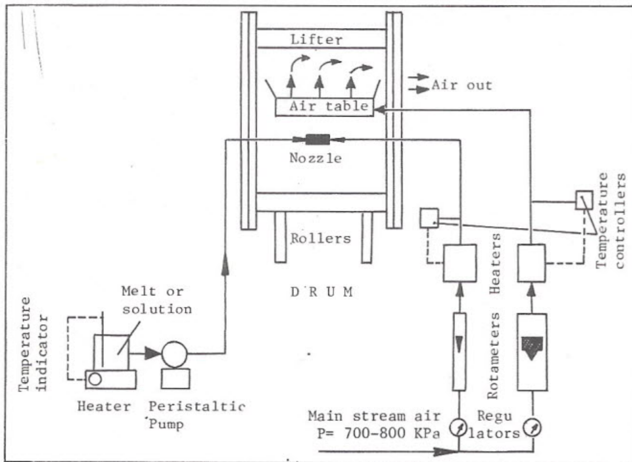


Figure 2. Schematic of the fluidised drum granulator.

The air table is used to enhance particle flow and dry or solidify the liquid coating. It is rectangular in shape, 100 mm wide and 160 mm long. The top of the air table is a perforated plate with 340 holes 1.25 mm in diameter, which act as the air distributor and contactor. The inlet pipe to the air table is set into the air table wind box to make sure that the air is distributed homogeneously along the air table length. Details of the air table design are shown in Figure 3.

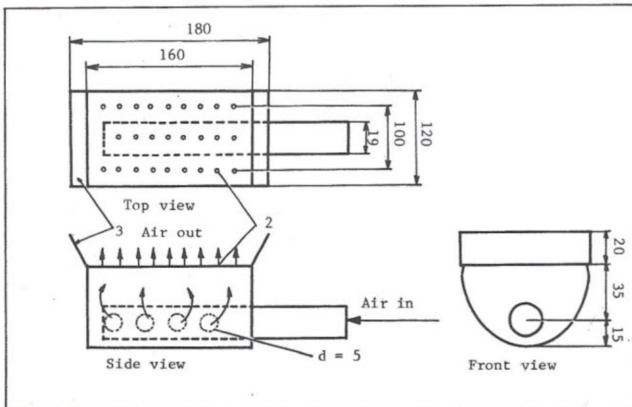


Figure 3. The schematic of the air table (scale in mm).
1. Air distributor
2. Air distributor/contactor
3. Flaps

As the length of the air table is slightly smaller than the drum length, metal flaps at each end of the table are used to channel particles falling from the ends of lifters on to the air table. The air table is positioned inside the drum such that the tumbling particles from the lifter will fall down to the air table area. No particles fall before or over the air table. The air table is set at an angle of 10° to ensure the particles flow freely along the air table.

The atomising nozzle is an internal mixing nozzle. It has six spray holes with 1.25 mm diameter, as shown in Figure 4. It is placed at the centre of the drum 5 cm beneath the air table and recessed 15 mm from the edge of the air table. The distance from the nozzle to the falling curtain of particles is about 50 mm. The nozzle sprays is horizontally, to make sure that the spray feed hits particle covering as wide an area as possible.

Compressed air at 700 kPa is supplied by the central air pipeline for the laboratories. The air to the air table passes through a pressure regulator and an oil/water filter as well as a rotameter (Metric 47G) enroute to an electric heater. All pressure gauges are Dobros type gauges ranging from 0 to 800 kPa. The air flow rate was between 0.01 - 0.03 standard m^3/s. The heater is constructed of 2 U-shaped thermorods (Helios heating) with a total capacity of 2 kW. The temperature of the air is controlled by a Shimaden SR10 controller. 3 K-type thermocouples are used to measure the temperatures of the air after leaving the heater before the inlet to the air table.

A separate air line is used to supply air to the atomising nozzle. The air is also heated to increase the temperature to 150°C (measured just after the heater). The heater is constructed of U-shaped thermorods with the capacity of 1 kW. The temperature of the air is controlled by MCS-Shiho temperature controller. The air flow rate was measured by Precision Bore flow meter Tube (F&P). The flow is between 0-100 l/min. The air flow rate was calibrated using a flow meter measuring to 0.01 l (RITTER, Bochum-Langendreer, Jerman).

In slurry mixing and spraying system wax is heated to 150-160°C in a 500 ml glass beaker placed on the heating plate. The molten wax temperature is monitored by a thermometer. The molten wax is pumped by a Peristaltic pump (Cole Palmer Masterflex 7015) through silicon tubing (diameter of 1/4") to the spray nozzle and meets the hot air in the outlet of the nozzle (Figure 4).

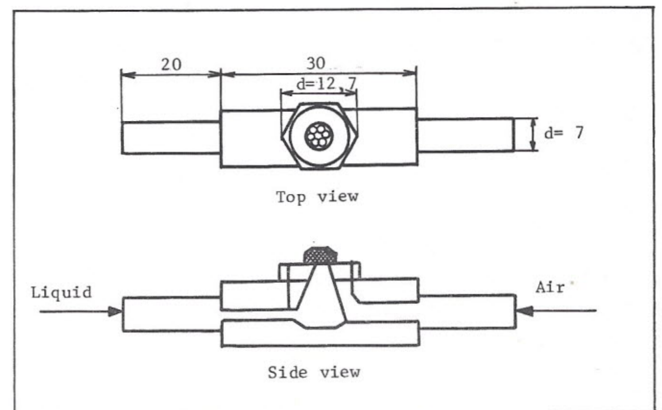
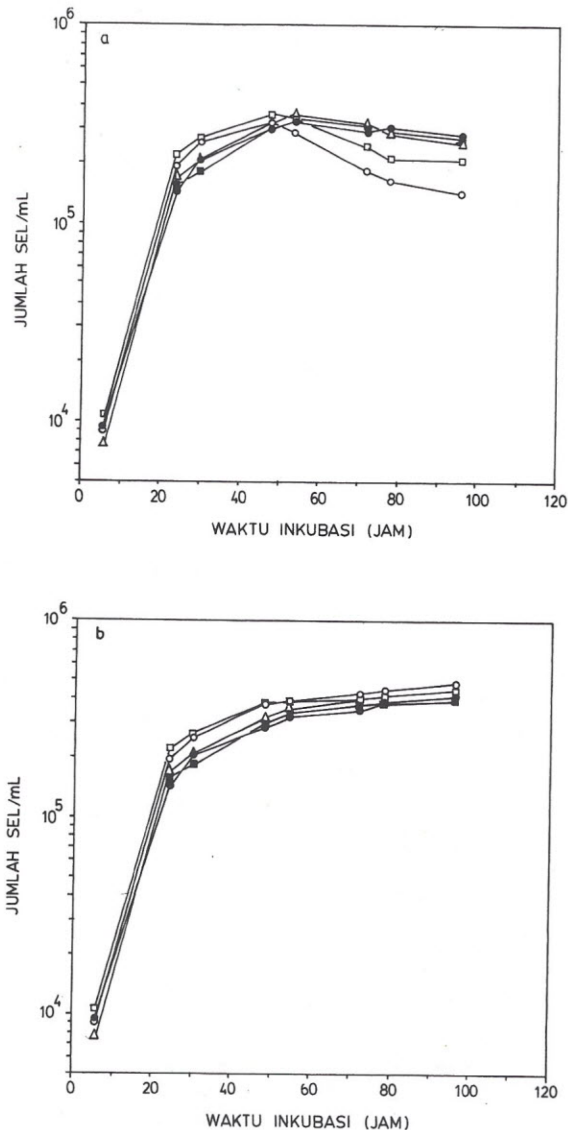


Figure 4. The schematic of the nozzle (scale in mm)

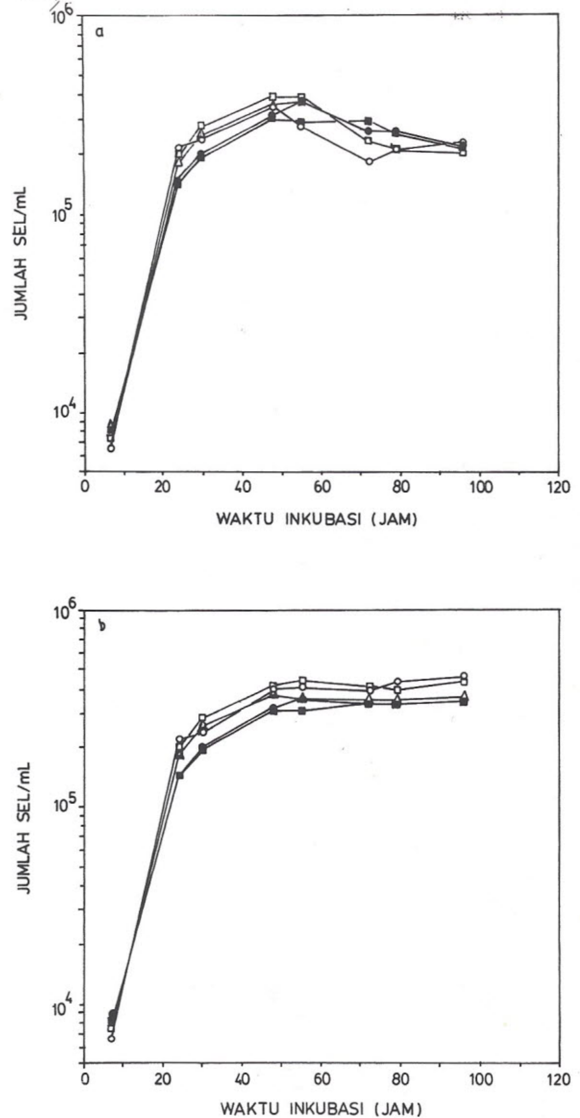
The pump has a solid state multiple speed controller to control the flow rate of the slurry. The flow rate range is between 17.5 and 45 g/min.

Hasil yang serupa diperoleh jika residu heksan (RH) ditambahkan ke dalam medium pertumbuhan protozoa. Kadar 0,001 - 0,01% RH dalam medium selama 96 jam inkubasi tidak menunjukkan pengaruh yang nyata terhadap populasi sel protozoa (P>0,05). RH kadar 0,1–1 % dalam medium menunjukkan kenaikan sel total protozoa yang nyata (P<0,05) dibandingkan blanko (Gambar 3b). Akan tetapi dari sel total ini, setelah waktu inkubasi 54 jam, banyak sel yang mati sehingga mempengaruhi jumlah sel hidup dan terlihat adanya hambatan. Hambatan yang nyata terlihat dalam medium yang mengandung 1 % RH. Residu heksan (1%) menyebabkan hambatan pertumbuhan protozoa sekitar 12,9% (54 jam) sampai 49,1% (96 jam), (Gambar 3a).



Gambar 3. Kurva pertumbuhan *T.pyriformis* GL dalam medium pertumbuhan yang mengandung residu heksan:
(a). pertumbuhan dinyatakan dengan perubahan jumlah sel hidup di dalam medium,
(b). pertumbuhan dinyatakan dengan perubahan jumlah sel total di dalam medium.
(●) tanpa residu heksan, (■) dengan residu heksan 0,001 %, (Δ) dengan residu heksan 0,01 %, (□) dengan residu heksan 0,1 % dan (○) dengan residu heksan 1 %.

Pengaruh residu saga (RS) terhadap pertumbuhan *T.pyriformis* terlihat pada Gambar 4a dan 4b. Sel total protozoa bertambah banyak dengan makin besarnya kadar residu saga dalam medium. Terlihat bahwa RS tidak mempengaruhi populasi sel hidup protozoa. Jumlah sel hidup protozoa dalam medium yang mengandung RS (0,001- 1%) tidak berbeda nyata dibanding dengan blanko (P>0,05)



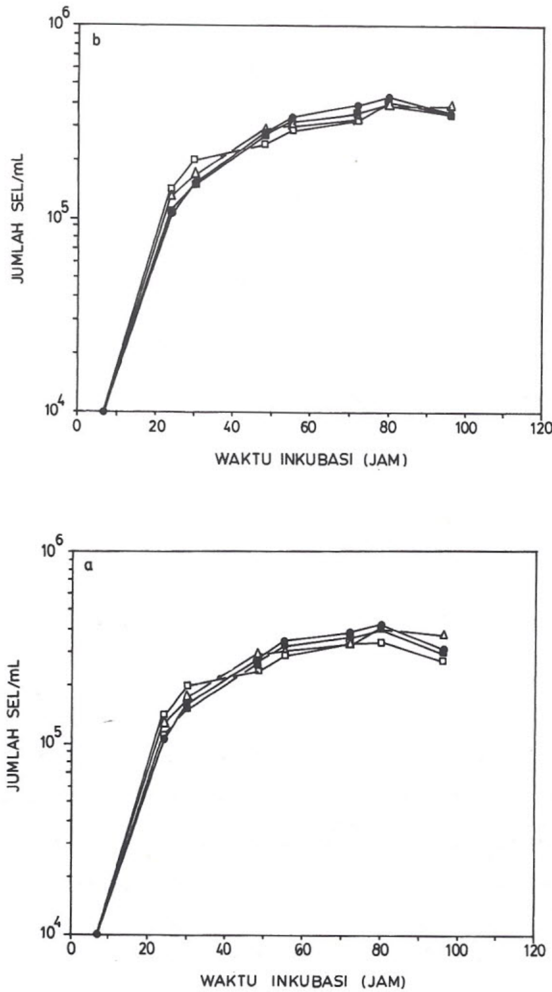
Gambar 4. Kurva pertumbuhan *T.pyriformis* GL dalam medium pertumbuhan yang mengandung residu saga:
(a). pertumbuhan dinyatakan dengan perubahan jumlah sel hidup di dalam medium,
(b). pertumbuhan dinyatakan dengan perubahan jumlah sel total di dalam medium.
(●) tanpa residu saga, (■) dengan residu saga 0,001% (Δ) dengan residu saga 0,01%, (□) dengan residu saga 0,1 % dan (○) dengan residu saga 1%.

Pengaruh hambatan keping biji saga dan residu heksan terhadap *T.pyriformis* menunjukkan gambaran yang serupa, seperti yang terlihat pada Gambar 2 dan 3. Residu heksan menunjukkan hambatan yang lebih kecil dibandingkan keping biji saga. Hal ini dapat disebabkan oleh proses ekstraksi keping biji saga dengan n-heksana, dimana kemungkinan beberapa senyawa yang aktif terhadap protozoa terekstraksi oleh n-heksana. Demikian juga dengan residu

saga yang dapat dikatakan tidak mempengaruhi pertumbuhan *T.pyriformis*. Ekstraksi residu heksan dengan etanol, diduga mengekstraksi senyawa aktif di dalamnya menyebabkan residu saga tidak aktif terhadap *T.pyriformis*. Hal ini dapat terlihat pada uji aktivitas minyak saga dan ekstrak etanol terhadap protozoa.

Pengaruh minyak saga terhadap pertumbuhan protozoa

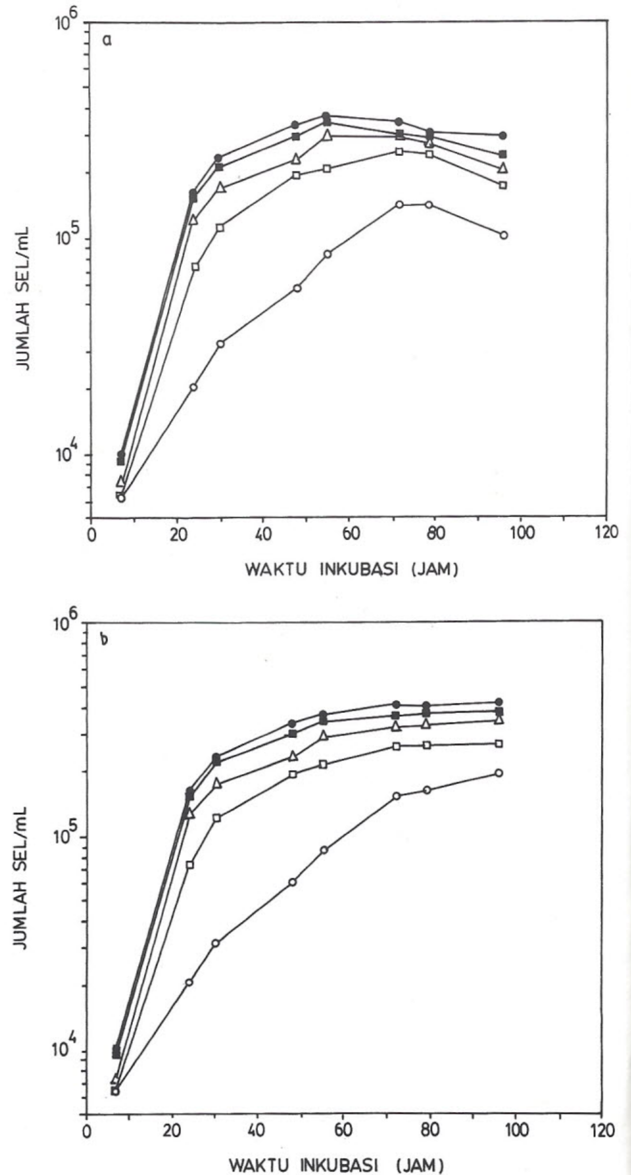
Pada uji aktivitas minyak saga terhadap *T.pyriformis*, ditambahkan 0,01 % *Cremophore EL* (BASF), suatu emulsifier sehingga minyak saga dapat terdispersi homogen dalam medium pertumbuhan. Minyak saga dalam kadar 0,001-0,1 % tidak menunjukkan pengaruh yang nyata terhadap pertumbuhan protozoa seperti terlihat pada Gambar 5a dan 5b. Ada kecenderungan baik jumlah sel total maupun sel hidup berkurang dengan bertambahnya kadar minyak saga dalam medium, tapi pengaruhnya tidak nyata ($P>0,05$).



Gambar 5. Kurva pertumbuhan *T.pyriformis* GL dalam medium pertumbuhan yang mengandung minyak saga:
(a). pertumbuhan dinyatakan dengan perubahan jumlah sel hidup di dalam medium.
(b). pertumbuhan dinyatakan dengan perubahan jumlah sel total di dalam medium.
(●) tanpa minyak saga, (■) dengan minyak saga 0,001%, (Δ) dengan minyak saga 0,01 %, (□) dengan minyak saga 0,1 %.

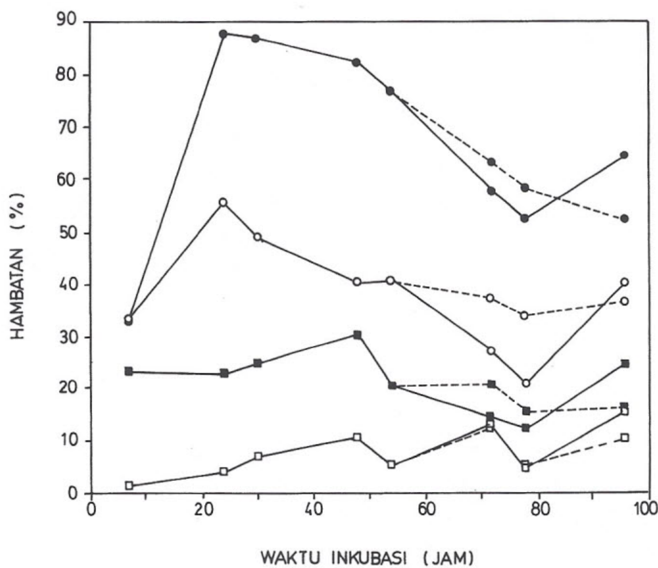
Pengaruh ekstrak etanol saga terhadap pertumbuhan protozoa

Pengaruh ekstrak etanol saga (EEt) terhadap *T.pyriformis* sudah terlihat nyata pada 7 jam inkubasi ($P<0,05$). Jumlah sel protozoa dalam medium yang mengandung 0,1 % dan 1 % EEt, lebih sedikit dibandingkan blanko. Mulai 7 jam sampai 96 jam inkubasi, pengaruh terhadap populasi sel total maupun sel hidup *T.pyriformis* terlihat nyata dalam medium yang mengandung 0,1 - 1 % EEt (Gambar 6a dan 6b). Jumlah selnya berkurang dibandingkan blanko sehingga terlihat adanya hambatan.



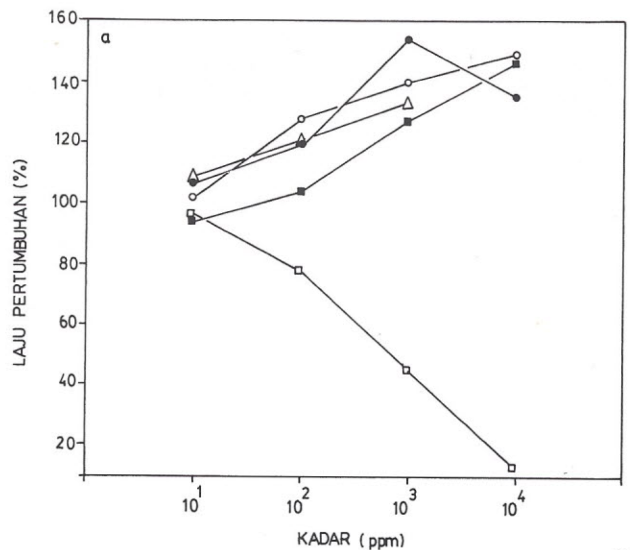
Gambar 6 Kurva pertumbuhan *T.pyriformis* GL dalam medium pertumbuhan yang mengandung ekstrak etanol biji saga:
(a). pertumbuhan dinyatakan dengan perubahan jumlah sel hidup di dalam medium,
(b). pertumbuhan dinyatakan dengan perubahan jumlah sel total di dalam medium.
(●) tanpa ekstrak etanol saga, (■) dengan ekstrak etanol saga 0,001 %, (Δ) dengan ekstrak etanol saga 0,01 %, (□) dengan ekstrak etanol saga 0,1 % dan (○) dengan ekstrak etanol saga 1%.

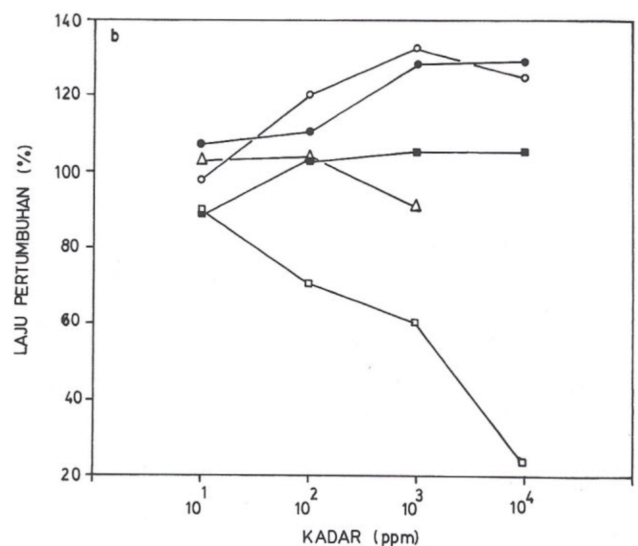
Dibandingkan dengan ekstrak-ekstrak biji saga lainnya, ekstrak etanol saga lebih jelas pengaruh hambatannya terhadap populasi *T.pyriformis*. Pengaruh ekstrak etanol saga sudah nyata mulai 7 jam inkubasi dibandingkan ekstrak-ekstrak lainnya, yang baru terlihat setelah 48 jam atau 55 jam inkubasi. Dapat dikatakan hambatan ekstrak etanol lebih efektif mengingat pengaruhnya nyata pada fasa logaritma, dimana protozoa sedang berkembang biak. Hambatan ekstrak etanol saga terhadap sel total dan sel hidup *T.pyriformis* terlihat pada Gambar 7. Untuk kadar 0,01 % ekstrak etanol saga, hambatan maksimum teramati pada 48 jam inkubasi sebanyak 29,9%, sedangkan untuk kadar 0,1 % dan 1 % ekstrak etanol saga hambatan maksimum teramati pada 24 jam inkubasi sebanyak 55,1% dan 87,6%.



Gambar 7. Hambatan ekstrak etanol saga terhadap populasi *T.pyriformis* GL.
- terhadap populasi sel hidup
-- terhadap populasi sel total
(□) dengan 0,001 % ekstrak etanol saga
(■) dengan 0,01 % ekstrak etanol saga
(○) dengan 0,1 % ekstrak etanol saga
(●) dengan 1 % ekstrak etanol saga

Hambatan ekstrak etanol juga teramati pada laju pertumbuhan *T.pyriformis* (persentase jumlah sel terhadap blanko) dalam medium pertumbuhan yang mengandung ekstrak-ekstrak saga, seperti terlihat pada Gambar 8a dan 8b. Hanya ekstrak etanol memperlihatkan laju pertumbuhan yang makin kecil dengan bertambah besarnya kadar, sedangkan ekstrak-ekstrak lainnya memperlihatkan laju pertumbuhan yang lebih besar dari 100% selama 24 jam dan 48 jam waktu inkubasi. Sehingga dapat disimpulkan bahwa dalam penapisan sifat antiprotozoa dalan biji saga, ekstrak etanol biji saga menunjukkan sifat antiprotozoa yang paling besar dibandingkan ekstrak lainnya. Hasil perhitungan ED50 (kadar ekstrak etanol yang menyebabkan hambatan 50%) menurut metoda log probit Miller dan Tainter (17) adalah 700 ppm.

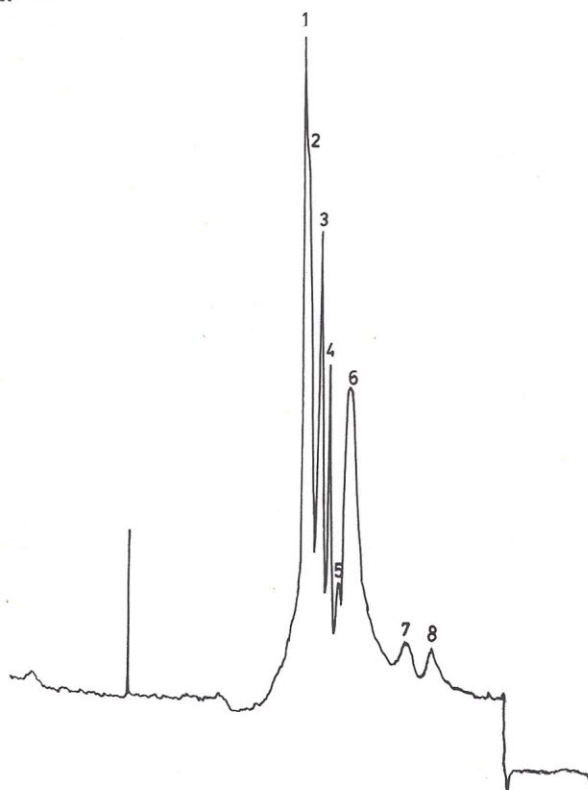




Gambar 8. Laju pertumbuhan (persen jumlah sel terhadap blanko) *T.pyriformis* dalam medium pertumbuhan yang mengandung ekstrak-ekstrak saga.
(a) waktu inkubasi 24 jam
(b) waktu inkubasi 48 jam
(■) keping saga
(Δ) minyak saga
(●) residu heksan
(○) residu saga
(□) ekstrak etanol

Analisis lebih lanjut dilakukan terhadap ekstrak etanol saga, mengingat aktivitasnya terhadap *T.pyriformis* lebih besar dibandingkan ekstrak-ekstrak lainnya dan sebagai pendahuluan untuk isolasi senyawa anti protozoa dalam biji saga. Analisis kualitatif fitokimia terhadap saponin, alkaloid, steroid, fenol, tanin dan terpenoid menunjukkan hasil positif terhadap saponin dan alkaloida. Analisis dengan kromatografi cair kinerja tinggi menghasilkan kromatogram seperti terlihat pada Gambar 9. Diamati sekitar 8 puncak kromatogram dengan spesifikasi seperti tertera pada

Tabel 1. Dari hasil kromatografi terlihat bahwa ekstrak etanol saga masih terdiri atas campuran paling sedikit delapan komponen. Fraksinasi lebih lanjut perlu dilakukan untuk mengisolasi senyawa toksis/antinutrisi dalam biji saga.



Gambar 9. Kromatogram ekstrak etanol saga pada kolom RP18, eluen metanol: air = 8:2 (v/v). Kecepatan alir 0,7 ml/menit. Detektor UV, λ : 313 nm.

Tabel 1. Puncak-puncak kromatogram ekstrak etanol saga

	Waktu retensi (menit)	komposisi * (%)
1.	3,80	26,05
2.	3,90	21,49
3.	4,10	18,44
4.	4,35	13,25
5.	4,55	4,48
6.	4,80	12,18
7.	6,05	2,14
8.	6,55	1,97

* perbandingan tinggi puncak

KESIMPULAN

Dari hasil uji aktivitas antiprotozoa ekstrak ekstrak biji saga dapat disimpulkan :

1. Keping biji saga menunjukkan sifat anti protozoa terhadap *T.pyriformis* GL.
2. Dari hasil ekstraksi biji saga, ekstrak heksana (minyak saga) pada kadar sampai 0,1% dalam medium pertumbuhan tidak menunjukkan pengaruh yang nyata terhadap pertumbuhan *T.pyriformis* GL. Sedangkan ekstrak etanol biji saga dalam medium pertumbuhan menunjukkan hambatan yang nyata terhadap *T.pyriformis* GL. Pada 24 jam waktu inkubasi, 0,1% ekstrak etanol saga menghambat pertumbuhan *T.pyriformis* GL sebanyak 55,1% dan 1% ekstrak etanol saga menunjukkan hambatan sebanyak 87,6%. Sifat antiprotozoa ekstrak etanol saga adalah yang paling besar dibandingkan ekstrak lainnya.
3. Analisis kualitatif ekstrak etanol saga menunjukkan adanya saponin dan alkaloid. Hasil analisis dengan kromatografi cair kinerja tinggi didapatkan paling sedikit delapan komponen dalam ekstrak etanol saga.

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