Optimum feed- and tank-operating conditions to maintain constant particle concentration in feed streams of particulate fluid food suspensions

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Summary
Ensuring product safety and constant quality in aseptic processing of particulate fluid foods can be a problem if feed particle concentration varies with time. The variability of particle concentration distributions from a vertical stirred feed tank was measured as standard deviations of mean particle concentrations at the tank outlet and as differences between this concentration and the bulk concentration in the tank. The influence of some operating variables (volume of fluid in the tank, rotational speed of the agitating paddles, bulk particle concentration, liquid viscosity and flow rate) was studied using a factorial experimental design at two levels. An operating chart was drawn, using response surface analysis for selecting the optimum volume of fluid in the tank as a function of particle concentration and fluid viscosity.

Keywords
Aseptic processing, particle distributions in fluid suspension, residence time distribution, stirred tank operating parameters.

Introduction
The assessment of aseptic processing of particulate fluid foods involves several factors from two broad types: heat transfer and residence time, as suggested by Heldman (1989). However, in order to ensure product safety without unnecessary overprocessing and detrimental quality, the constancy of distribution of the particles in the feed mixture entering the system is particularly important.

Dignan et al. (1989) considered potential health hazards and suggested that the fastest particle travelling in the relevant parts of the system is of prime importance in aseptic processing. This implies that the heat treatment of each particle is primarily dependent on its residence time. Such an assumption may not be applicable if the feed includes particles with different characteristics such as size, geometry and thermal properties. In this situation the fastest particle may not be the one with the lowest heat treatment and a more detailed study may be necessary. Actual identification of
the fastest particle may be possible but in practice this can be overcome by considering
the residence time distribution of the particles.

In recent years several investigations related to the residence time distribution
of particulate foods have been carried out on scraped-surface heat exchangers or
holding tubes; they were reviewed by Singh & Lee (1992). Berry (1989) predicted
statistically the residence time distribution of the fastest particle, and Sastry (1986),
McCoy et al. (1987) and Yang & Swartzel (1992) determined residence time distribution
of particles in holding tubes. As only single particles were used in these studies,
there was no variability in the feed concentration, despite its paramount importance
in full scale industrial processes. Sastry and Zuritz (1987) developed a mathematical
model for the prediction of particle trajectories and velocities in tubes, but again,
due to the complexity of the problem, variables and forces involved, only single
particles were used and interactive effects between particles were not considered.
Taeymans et al. (1986) used tanks to feed three cylindrical scraped-surface heat
exchangers, 158 mm in diameter and 508 mm in length. The solid particles were
6-mm diameter alginate beads and the concentration in the fluid (water) was 4%.
The variance of the particle concentration at the outlet of the system was checked
and considered negligible (a standard deviation of ± 0.21 was calculated from the
author's data). More recently Palmieri et al. (1992) used a 200-L tank with a paddle
agitator to feed 10-mm cubes of Bintje cultivar potatoes in a fluid of similar density
to a holding tube of 50 mm internal diameter. The variation in particle distribution
was not measured or discussed.

In residence time studies of aseptic processing of particulate fluid foods the
importance of the constancy of system input feed is rarely considered and never
analysed systematically. Residence time distribution curves generated from several
runs of individual particles are useful to discuss residence time distribution issues but
the important industrial processing aspects of bulk particle concentration variation
and between-particle interactions are missing.

In chemical engineering effective mixing of solids with liquids is a known problem
even with finely divided solid particles (Foust et al., 1980; Badger & Banchero,
1985). However, with foods, where particles have dimensions of several mm, the
problem is much more complex and, in general, liquid-mixing equipment is not
adequate (McCabe, 1985). Fluidization (Davidson et al., 1985) could be considered
for coarse (ϕ > 1 mm) particles, e.g. as in fluidized bed combustion of coal, or larger
(9 mm) glass spheres (Coulson & Richardson, 1978). In practice the use of fluidized
beds for feeding large continuous aseptic processing systems would require complex
design and operating control parameters, and would be very expensive.

The main objective of this work was to evaluate the influence of operating
variables on simulated food particle concentration and distribution in a vertical
stirred feeding tank. The operating variables considered were: volume of liquid (V),
bulk particle concentration (C), rotational speed of the agitating paddles (ω) in the
feed tank, liquid viscosity (μ) and flow rate (Q). The final objective of this work
was to establish optimum operating conditions to minimize the variability in feed
concentration.

Materials and methods
Spherical polystyrene particles (Hoover Precision Products, Inc.) with 9.52 ± 0.02 mm
diameter and 1.065 ± 0.015 g cm⁻³ density were used as model food particles. Sodium
Table 1. Shear-stress characteristics* of the carboxymethylcellulose (CMC) solutions at 20°C

<table>
<thead>
<tr>
<th>% CMC</th>
<th>Behaviour</th>
<th>K (Pa sⁿ)</th>
<th>n</th>
<th>Flow behaviour index</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>Newtonian</td>
<td>0.0010</td>
<td>1.00</td>
<td></td>
</tr>
<tr>
<td>0.10</td>
<td>Newtonian</td>
<td>0.0021 ± 0.0003</td>
<td>1.02 ± 0.02</td>
<td></td>
</tr>
<tr>
<td>0.20</td>
<td>Pseudoplastic</td>
<td>0.021 ± 0.001</td>
<td>0.83 ± 0.01</td>
<td></td>
</tr>
<tr>
<td>0.30</td>
<td>Pseudoplastic</td>
<td>0.064 ± 0.004</td>
<td>0.75 ± 0.01</td>
<td></td>
</tr>
</tbody>
</table>

* shear stress = K (shear rate)ⁿ

carboxymethylcellulose (CMC) solutions were prepared by slowly adding to water at 60°C, with continuous agitation, maintained for at least 15 h to give a density independent of concentration in the range used (1.00 ± 0.01 g cm⁻³). This was used as a model fluid food because it provides Newtonian and pseudoplastic rheological characteristics with different concentrations (Table 1).

The aseptic processing system used (Fig. 1) was a liquid feed tank (A), a solid/liquid mixture feed tank (B), a pump (C), a visualization section (D) and a tubular (22-mm i.d.) aseptic processing unit with heating, holding and cooling sections (E₁, E₂ and E₃).

The vertical, 180-L, cylindrical feed tank (0.60 m d, 0.65 m maximum depth) had a bottom slope of 5°, incorporating a vortex-breaking cone (0.20 m d × 0.135 m high) fixed to the axis of the bottom of the tank, centre 0.105 m from the tank centre (Fig. 2). Two three-bladed marine propellers (d 0.20 m) on a shaft at 37° from the vertical were used in an off-centre position in the unbaffled tank.

The feed outlet was 50 mm d in the lateral lower position of the feed tank and the feed inlet was into the open top of the tank.

The pump (developed specifically for this work by ARSOPI S.A., Vale de Cambra, Portugal) was single rotor with nine rubber blades suitable for particles that should not be crushed (e.g. peas, beans, or the model particles used in this work).

Procedure

The feed tank B was filled with the solid/liquid mixture up to the specified volume and stirred. Tank A was filled with liquid only which was pumped through the
system until all air was purged. Recirculation was changed from tank A to B and maintained for sufficient time to stabilize the system and videotape the particle flow in the transparent section.

The concentration at the tank outlet was determined from the number of particles passing the transparent section in 24 random time-interval 5-s periods, counted from the videotape from the moment the system was stable until a maximum of 12 min. Two parameters were used to evaluate the constancy of the concentration: difference of average concentrations and coefficient of variation.

Difference of Average Concentrations (DAC) represents the percentage difference between the average concentration at the tank outlet \((C_0^{av})\), based on the analysis of the videotape over the time period of the 24 samples taken and the bulk concentration in the feed tank \((C)\), based on volume of particles added to the specified volume of liquid in tank B:

\[
DAC = \frac{C_0^{av} - C}{C} \times 100
\]

where:

\[
C_0^{av} = \frac{\sum_{i=1}^{24} C_0}{24}
\]

If the DAC parameter is negative, the bulk concentration in the tank is higher than the average concentration at the outlet and therefore solids are accumulating in the tank; if it is positive, the reverse is happening. Inevitably, a DAC parameter different from zero would lead to significant variations somewhere along the operation.

The other parameter, the Coefficient of Variation (CV), corresponds to the percentage standard deviation of the outlet concentration \((C_0)\) normalized with the average outlet concentration:
The operating variables were: volume of fluid (V) in tank B, bulk particle concentration (C), rotational speed of the agitating paddles (ω) in the feed tank, liquid viscosity (µ) and flow rate (Q). There were two sets of experiments. The first was a 2-level factorial design to identify the individual and joint effects of the variables to be tested. The second was a 4-level factorial design, to define optimal operating conditions for the chosen variables using response surface methodology. All the experiments were carried out at room temperature (20°C).

Individual and interactive effects

In order to determine the effect of all variables and combinations of variables on the parameters chosen, a 2⁵ factorial design was used (Box et al., 1978). This statistical method is commonly used in food science and technology in areas such as drying, extrusion, extraction, sensory analysis and microorganism activity and growth, e.g. Lacroix & Lachance (1988) and Bains & Ramaswamy (1989). Two sets of 32 experiments were carried out randomly with all possible combinations of minimum and maximum levels in each variable (Table 2). The two sets allowed for the calculation of the standard deviation.

The viscosity levels were achieved by adjusting the CMC concentration. Water is a Newtonian fluid and CMC solutions are non-Newtonian fluids with a behaviour dependent on the CMC concentration. The viscosity of CMC solutions varies with the particular batch of CMC and therefore the rheological behaviour of each solution was measured with a coaxial cylinder viscometer (Contraves RHEOMAT Model 115, Contraves AG, Zurich, Switzerland). The data were fitted to a power law model (Table 1). Viscosity increased significantly with the addition of CMC, but Newtonian behaviour was still evident at the lower concentrations, with the solution becoming more pseudoplastic as CMC concentration increased. The fluid consistency index (that is, the apparent viscosity at a unit shear rate) could have been used as a variable, but the change in rheological behaviour would not have been apparent in those values. Instead both the fluid consistency index and the CMC concentration represented the viscosity variable throughout this work. To simplify, the term µ in the text and equations refers to CMC concentration.

Operating chart

Based on the preliminary conclusions drawn from the factorial design analysis, further experiments were designed in order to identify the best way to determine

Table 2. Level of variables used in the 2⁵ factorial design

<table>
<thead>
<tr>
<th>Variable level</th>
<th>Volume of fluid (L)</th>
<th>Baffle speed (r.p.m.)</th>
<th>Particle concentration (% v/v)</th>
<th>Liquid viscosity (PA sⁿ/% CMC)</th>
<th>Flow rate (L h⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>−</td>
<td>80</td>
<td>93</td>
<td>1.0</td>
<td>0.0010/0.00</td>
<td>286</td>
</tr>
<tr>
<td>+</td>
<td>120</td>
<td>116</td>
<td>3.1</td>
<td>0.064/0.30</td>
<td>375</td>
</tr>
</tbody>
</table>
optimum operating conditions. The first experiments showed that some variables were optimum at one extreme (maximum) while others had no significant effect. These preliminary experiments showed that the DAC parameter was the most sensitive, with an optimum close to the CV parameter optimum. Therefore, an operating chart for choosing the optimum conditions was drawn considering the DAC parameter only. The CV parameter was calculated for each node of the chart, ensuring that the choice could be done on a DAC basis only. The three variables involved in this study were the volume of liquid in the feed tank, fluid viscosity and particle concentration. A 4-level factorial design (Table 3) was then used, involving 64 experiments, also randomly performed.

An operating chart was then developed using a response surface methodology (Gacula and Singh, 1984; Box et al., 1978). In the food field, this methodology has been used particularly in sensory analysis, e.g. Abdullah et al. (1993) and extrusion studies, e.g. Olkku et al. (1984). The predictive equations were obtained from the data using the Statistics/Data Analysis (STATA 2.1, Computing Resource Center, 1990) software for multiple regression analysis. First and second-order models could be considered. A second-order model was used because first-order models often lead to inadequate results and poor response surfaces. Abdullah et al. (1993) reported that if the predictive equations have a $R^2$ ($R =$ regression coefficient) equal to or greater than 0.70 they are adequate to generate contour plots.

Results and discussion

Individual and interactive effects

The analysis of the individual or interactive effects of the variables can be performed by considering the normal plot of the effects, for each parameter considered, based on the results of the factorial design. If no significant effect exists it is possible to draw a straight line through all the points. Points away from this line represent a significant variable, or combination of variables, identified as falling outside the calculated 95 or 99% confidence region. The ANOVA procedure of the STATVIEW software (Statview, Abacus Concepts, Inc., 1992) was used for these calculations.

Analysis of the % difference between the average tank outlet concentration and bulk concentration in the feed tank – DAC

The volume of fluid in the tank (V) and the liquid viscosity ($\mu$) have the most significant individual effects (Fig. 3), but the combined effect of concentration and

<table>
<thead>
<tr>
<th>Variable level</th>
<th>Volume of fluid (L)</th>
<th>Particle concentration (% v/v)</th>
<th>Liquid viscosity (Pa s%/CMC)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>80</td>
<td>1.0</td>
<td>0.0010/0.00</td>
</tr>
<tr>
<td>2</td>
<td>90</td>
<td>1.7</td>
<td>0.0021/0.10</td>
</tr>
<tr>
<td>3</td>
<td>100</td>
<td>2.4</td>
<td>0.021/0.20</td>
</tr>
<tr>
<td>4</td>
<td>110</td>
<td>3.1</td>
<td>0.064/0.30</td>
</tr>
</tbody>
</table>

* Rotational speed of the agitating baffles = 93 r.p.m. flow rate = 375 L h$^{-1}$
Constant particulate feed concentrations effect

viscosity (C/μ) was also significant. These were the only points that fell outside the 99% confidence region. The rotational speed of the paddles and the flow rate had no significant effect.

The magnitudes (Fig. 4) of individual effects are represented by straight lines and of double effects by square edges. Bold lines represent the magnitude of the variation of the parameter. The more significant the effect, the longer the line. A line drawn from left to right or from bottom to top indicates a positive effect, that is, an increase in the parameter, while a line drawn from right to left or from top to bottom indicates a negative effect, that is, a decrease in the parameter. The − sign represents the low level of the variable and the + sign the high level. Small numbers indicate the value of the parameter on that corner and the large numbers the variation in the parameter from one corner to another (that is, the difference between the parameter for the variable at the + level and at the − level).

The DAC parameter may be positive or negative. Optimal operating conditions would be those that would lead to a DAC as close to zero as possible.

Several conclusions may be drawn from Fig. 4. When the volume of liquid in the feed tank is low there is an accumulation of particles in the tank and the outlet particle concentration is lower than the tank bulk concentration (the DAC parameter is

Figure 3. Normal plot of the % difference of average concentrations for the 25 factorial design; the frequency distribution is shown at the bottom of the plot.
As the volume is increased the accumulation is reduced and after a certain volume this phenomenon is even reversed. Viscosity also has a similar effect, more pronounced for low particle concentration, as can be seen from the analysis of this combined effect. Fluids with a higher concentration of particles present fewer problems (the DAC parameter is reduced), particularly for less viscous fluids. The influence of concentration arises only when linked to viscosity and not on its own. To summarize, agitation speed and flow rate are not very relevant in the range considered, while for viscosity, volume of liquid in the tank and particle concentration an optimum combination of these variables should be found.

Analysis of the coefficient of variation – CV

Similar plots to those for the DAC parameter are given for CV (Figs 5 and 6). CV is always positive and ideally should be zero. All the variables except the rotational speed of the agitating paddles have, to some extent, individual effects (Fig. 5). The particle concentration (C), the volume of fluid in the tank (V), the viscosity (μ) and the combined effect volume/concentration (V/C) have the greatest influence, falling outside the 99% confidence region. Additional effects that fall outside the 95% confidence region are flow rate (Q), flow rate/particle concentration (Q/C) and volume of fluid in the tank/viscosity (V/μ).

An increase in any variable always results in a decrease in the CV parameter (Fig. 6). The effect of the volume of liquid in the feed tank is more important for low viscosity fluids and for low particle concentrations. An increase in flow rate also has a more significant effect for low particle concentration, being negligible for high concentrations. Feeds with a higher particle concentration and high viscosity present fewer problems, specially if the volume of fluid in the tank is low.

Operating Chart

Using the coefficient of variation as a criterion, the best operating conditions correspond to the use of maximum levels of all variables. The flow rate does not affect the DAC parameter and therefore should be set at the maximum level. Stirring
Constant particulate feed concentrations

\[ \frac{\text{V}}{\text{C}} \approx 0.9 \]

\[ \frac{\text{V}}{\text{C}} \approx 0.4 \]

10

5

14

9

21

26

20

19

17

16

15

13

12

11

\[ \text{Effect no.:} \]

1 - \( \frac{\text{C}}{\text{V}} \)

2 - \( \omega \frac{\text{V}}{\mu} \)

3 - \( \frac{\text{V}}{\text{Y}} \)

4 - \( \frac{\text{Y}}{\mu} \)

5 - \( \frac{\text{Q}}{\text{V}} \)

6 - \( \frac{\text{V}}{\text{C}} \)

7 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

8 - \( \omega \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

9 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

10 - \( \frac{\omega}{\text{Q}} \frac{\text{V}}{\mu} \)

11 - \( \frac{\omega}{\text{Q}} \frac{\text{C}}{\mu} \)

12 - \( \omega \frac{\text{V}}{\mu} \)

13 - \( \frac{\text{Q}}{\mu} \)

14 - \( \frac{\omega}{\text{Q}} \frac{\text{V}}{\mu} \)

15 - \( \frac{\omega}{\text{C}} \frac{\text{V}}{\mu} \)

16 - \( \frac{\omega}{\text{Q}} \frac{\text{V}}{\mu} \)

17 - \( \frac{\omega}{\text{Q}} \frac{\text{V}}{\mu} \)

18 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

19 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

20 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

21 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

22 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

23 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

24 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

25 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

26 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

27 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

28 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

29 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

30 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

31 - \( \frac{\text{Q}}{\text{V}} \frac{\text{C}}{\mu} \)

Figure 5. Normal plot of the coefficient of variation for the \( 2^5 \) factorial design; the frequency distribution is shown at the bottom of the plot.

does not have a significant influence in most situations and therefore a minimum level can be used for energy efficiency. However, for a feed of given viscosity and particle concentration, although a maximum tank volume of fluid would decrease the outlet concentration standard deviation, the DAC suggests an intermediate value. If the DAC is not zero there is a positive or negative accumulation of particles in the tank, and inevitably instability would develop in the system.

Some experiments were carried out to assess the dependence of \( \text{CV} \) and DAC on the volume of fluid in the tank, for feeds with different characteristics (Figs 7 and 8); the results from DAC indicate optimal volumes between 80 and 100 L. As expected for low viscosity fluids and low particle concentration, \( \text{CV} \) decreased with increasing volume of fluid, but the effect was small in the 80—100 L range. This means that for most situations, optimal conditions may be defined only in terms of DAC. Therefore, a further set of 64 random experiments (four volumes of liquid × four particle concentrations × four liquid viscosities) was designed (Table 3), DAC calculated and the results regressed to give:

\[
\text{Y} = 4.307 + 14.243X_1 + 3.045X_2 + 0.322X_3 + 5.599X_4^2 - 1.171X_5^2 + 2.160X_6^2
- 3.343X_1X_2 - 10.486X_1X_3 - 4.143X_2X_3
\]

(4)
where $Y =$ DAC parameter; $X_1 = (V - 95)/15$; $X_2 = C - 2.0$ and $X_3 = (\mu - 0.15)/0.15$ with $R^2 = 0.74$.

Adequate contour plots were generated using this predictive equation. To draw the operating chart, four volumes (80, 90, 100 and 110 L) were fixed and for each, the experimental standard deviation limit lines corresponding to a DAC of $-6.8$ and $+6.8$ were constructed. The areas between these lines represent the combination of particle concentration and fluid viscosity for which the volume is optimum (Fig. 9). Results showed that in the viscosity and particle concentration ranges considered it was not possible to operate adequately with a volume of fluid higher than 100 L. As a result, no area is represented for such volumes in the operating chart. For some optimum conditions it is possible to operate with more than one volume (e.g. for $\mu = 0.15\%$ CMC and $C = 2.0\%$ v/v either 90 or 100 L would produce results) and there are two regions where it is only advisable to operate with one volume, one at 90 L (e.g. $\mu = 0.05\%$ CMC and $C = 2.4\%$ v/v) and another at 100 L (e.g. $\mu = 0.10\%$ CMC and $C = 1.0\%$ v/v). It should not be concluded that the volumes represented in the chart are restrictive. In regions where it is possible to operate at two different volumes it is probably also possible to operate at intermediate volumes and other lines of iso-volume of liquid could be drawn using the predictive equation. It was intended, however, to keep the chart simple to read. For points falling in a region...
where two or three volumes are suggested by the chart, if one limit line is further away than the other(s) the corresponding volume would be preferable (e.g. for $\mu = 0.15\%$ CMC and $C = 2.6\%$ v/v, 90 L would be the best option, while for $\mu = 0.12\%$ CMC and $C = 1.8\%$ v/v either 90 or 100 L would give identical results). For a small area, no volume gives a satisfactory result (e.g. $\mu = 0.02\%$ CMC and $C = 1.7\%$ v/v).

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**Figure 7.** Influence of the volume (V) of fluid in the tank on the % difference of average concentrations for two particle concentrations (C): (top) low viscosity fluids (water); (bottom) high viscosity fluids (0.30% CMC solution). Rotational speed of the agitating paddles = 116 r.p.m. Flow rate = 375 L h$^{-1}$. 

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Figure 8. Influence of the volume (V) of fluid in the tank on the coefficient of variation for two particle concentrations: (top) low viscosity fluids (water); (bottom) high viscosity fluids (0.30% CMC solution). Rotational speed of the agitating paddles = 93 r.p.m. Flow rate = 375 L h\(^{-1}\).

Conclusions
It was found that for the tank used and the range of operating conditions tested, maximum flow rate (375 L h\(^{-1}\)) should be used. A lower rotational speed of the agitating paddles (93 r.p.m) can be used. The volume of liquid in the tank should be
Chosen as a function of particle concentration and fluid viscosity and an operating chart was drawn to do this. Optimum volumes were 80, 90 and 100 L, with some combinations falling in areas where two or even all three volumes could actually be used. There was one small area, for low viscosity and particle concentrations between around 1.5–2%, where no optimum volume existed.

Since some variables are intrinsic to the tank design, these results cannot be applied directly to other tanks. Also, conclusions are only valid for the range of conditions tested. However, this work provides a case study and presents methodologies to expedite this situation.

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The authors thank Junta Nacional de Investigação Científica e Tecnológica and CEC (FLAIR Programme) for financial support, and acknowledge the invaluable support of ARSOPI, the metallurgical company where the aseptic processing system used in this work was designed and built. A special reference is given to Mr Armando Pinho and Mr Ernesto Ferreira for their constant support.
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