Robustness studies on coal gasification process variables

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Abstract
Optimisation of the Sasol-Lurgi gasification process was carried out by utilising the method of Factorial Experimental Design on the process variables of interest from a specifically equipped full-scale test gasifier. The process variables that govern gasification are not always fully controllable during normal operation. This paper discusses the application of statistical robustness studies as a method for determining the most efficient combination of process variables that might be hard-to-control during normal operation. Response surface models were developed in the process variables for each of the performance variables. It will be shown how statistical robustness studies provided the optimal conditions for sustainable gasifier operability and throughput. In particular, the optimum operability region is significantly expanded towards higher oxygen loads by changing and controlling the particle size distribution of the coal.

Key words: Gasification, response surface modelling, robustness studies

1 Introduction

The Secunda and Sasolburg plants in South Africa gasify approximately 30 million tons of bituminous coal per year to synthesis gas with an H₂/CO ratio of approximately 1.8, which is converted to fuels and chemicals via the Fischer-Tropsch process. A total of 97 Sasol-Lurgi fixed bed dry bottom gasifiers, 17 at Sasolburg and 80 at Secunda, have a combined production of approximately \(5.1 \times 10^6\) m³/h dry raw gas, which is equivalent to approximately \(3.6 \times 10^6\) m³/h pure synthesis gas. The Secunda plant is the largest syngas production facility of its kind in the world.

In 1998 Sasol decided to isolate one Sasol-Lurgi MK IV gasifier at the Secunda site as a test gasifier. This gasifier was equipped with additional instrumentation, which included a more sophisticated raw gas measurement and a dedicated coal feeding system. Several tests or experimental runs have been performed on the test gasifier during the last four years. The purpose of the tests was to further optimize product yields and to increase

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throughput. The tests were executed according to a full factorial experimental design [4]. Coetzer and Keyser (2003) developed statistical response surface models for evaluating the effect of the process variable changes, i.e. coal top size, coal bottom size, stone content (defined as the material that sinks at a relative density of 1.9), oxygen load (oxygen feed rate) and CO$_2$ in raw gas (RG) concentration, on gasifier performance in terms of carbon utilization and utility consumption.

However, the development of the models in [1] was based on the assumption that all the process variables are equally well controllable during normal operation. In this paper, the effect of the process variables on gasifier performance was evaluated when the assumption of fully controllable process variables cannot be made. Statistical process robustness studies were performed on the data set. Statistical robustness studies refer to the evaluation of process conditions for which the outputs or responses are insensitive to the variability transmitted from sources that are difficult or impossible to control during normal operation. Montgomery (1999) provided an explanation on how statistical robustness studies can be used for product and process design and development. In this paper, conditions of the process variables are evaluated through the application of statistical robustness studies that reduce the effect of the variability or instability of the hard-to-control variables on gasifier performance as measured by sustainable production and stable gasifier operation.

Two scenarios will be considered and evaluated; first it is assumed that the stone content of the coal is hard to control and the particle size distribution (PSD) of the coal is controllable, and secondly, it is assumed that the stone content of the coal is controllable but the PSD of the coal is hard to control. In practice the stone content can be highly variable in certain coal seams, and in such cases the stone content can be controlled by removing part or all of the stone in a beneficiation plant, which adds additional cost to the feedstock preparation and should therefore preferably be avoided. Manipulation and control of the coal PSD can be accomplished at a marginal cost by effective screening of the run-of-mine coal before feeding to the gasification process [1]. However, operational conditions can occur where PSD is hard to control.

Only two of the performance variables which affect sustainable gasifier performance will be discussed in this paper, namely pure gas yield (which is the volume of pure gas produced per mass of dry ash-free coal), and oxygen consumption (which is the volume of oxygen required to produce one unit volume of pure gas). The test gasifier data set is highly suitable for performing robustness studies, since the data were obtained through a statistically designed experimental program. The paper is outlined as follows. First the methodology of statistical robustness studies will be presented. Thereafter, the process and performance variables used in the robustness studies will be discussed, followed by an explanation of the approaches employed in the evaluations. Some of the most significant results obtained, are presented.

2 The methodology of process robustness studies

Experiments are generally performed in industry to study or to evaluate the performance of a system or process. Montgomery (1999) presented a process by the general model in Figure 1. The process can be visualized as a combination of components, materials,
people, equipment, processes and other recourses that function collectively to transform a set of inputs into outputs described by one or more response variables. The process depicted in Figure 1 has \( p = k + r \) variables that can potentially affect the performance of the process. Variables \( x_1, x_2, \ldots, x_k \) are controllable variables that can be adjusted to or held at specific target values with satisfactory accuracy. Variables \( z_1, z_2, \ldots, z_r \) are either hard-to-control or uncontrollable during normal operation of the process, although they may be controlled for performing a specific experiment.

Gasification is also a process of transforming a set of inputs to outputs, such as pure gas yield or carbon utilization. Stone content, coal top size, coal bottom size, oxygen load and \( \text{CO}_2 \) in raw gas concentration were investigated as the variables that can potentially influence the process. There are, of course, many other variables that might influence the gasification process but considerable effort has been made to control those variables during the test runs. The experimental conditions of the process variables investigated were deliberately changed according to a full factorial experimental design [4]. Experiments for studying the performance of a process depicted in Figure 1 may have one or all of the following objectives [7]:

1. To determine which variables are most influential on the responses.

2. To determine where to set the influential controllable variables so that the responses are almost always near their desired target values.

3. To determine where to set the influential controllable variables so that the variability in the responses is small.

4. To determine where to set the influential controllable variables so that the effects of the hard-to-control variables on the responses are small.

![Figure 1: General model of a process.](image)

Statistical response surface models were developed previously to address objectives 1 and 2 above [1]. However, objectives 3 and 4 have not been addressed previously. Therefore,
the aim of this paper is to discuss and present, through statistical robustness studies, the effect of the process variables on gasifier performance, when the assumption of fully controllable variables cannot be made. A process is considered to be robust when it performs consistently on target and is relatively insensitive to variables that are hard to control. The term robustness studies refers to statistical designed experiments to develop or improve a process or product to be insensitive to variability transmitted to the outputs from sources that are difficult or impossible to control during normal operation. Hard-to-control variables are sometimes also referred to as noise variables or environmental variables.

Montgomery (1990, 1999) and Lucas (1994) discussed the development of robust processes through the use of statistically designed experiments and response surface methodology as opposed to the use of the methods of Taguchi (1986). Genichi Taguchi advocated using designed experiments for what he termed robust parameter design. He suggested highly fractionated factorial designs and other orthogonal arrays, along with some novel statistical methods, to solve the problem of achieving robust products and processes. Myers and Montgomery (1995) provided a detailed discussion of the use of factorial or fractional factorial designs, as well as response surface designs, in preference to the orthogonal arrays of Taguchi. They argued and showed that the orthogonal arrays of Taguchi often lead to experimental designs that are too large and that the use of signal-to-noise ratios has some statistical shortcomings. An improved way of performing robustness studies is to deploy a response surface methodology approach, known as the dual response surface approach. Myers (1991) explained that the response surface approach involves designing an experiment with the hard-to-control variables and the controllable variables in one array. The experimental design should at least allow the estimation of two-order interactions between the controllable and hard-to-control variables. With the dual response surface approach a response surface model is constructed for the process mean as well as for the process variance. Therefore, the two-order interactions between the controllable and hard-to-control variables will make up the terms in the response surface model for the process variance.

A response surface model in the controllable and hard-to-control variables for performing robustness studies is of the form:

$$y(x, z) = f(x) + h(x, z) + \varepsilon$$  \hspace{1cm} (1)

where

$$f(x) = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i<j=1}^{k} \beta_{ij} x_i x_j$$

and

$$h(x, z) = \sum_{j=1}^{r} \gamma_j z_j + \sum_{i=1}^{k} \sum_{j=1}^{r} \delta_{ij} x_i z_j.$$ 

Here $x_i, i = 1, 2, \ldots, k,$ are the controllable variables and $z_j, j = 1, 2, \ldots, r,$ are the uncontrollable variables. Furthermore $\beta_i, i = 1, 2, \ldots, k$ and $\beta_{ij}, i, j = 1, 2, \ldots, k, i \neq j,$ as well as $\gamma_j, j = 1, 2, \ldots, r$ and $\delta_{ij}, i = 1, 2, \ldots, k, j = 1, 2, \ldots, r,$ are parameters to be estimated, whilst $\varepsilon$ is an error term representing other sources of variability not accounted
for in \(y(x, z)\), such as measurement errors on the response. Model (1) contains the main effects and interactions of the controllable variables, the main effects of the hard-to-control variables and the interactions between the controllable and hard-to-control variables. Of course, second-order effects of the controllable and the hard-to-control variables may also be included in the model. A model such as (1) can be constructed on data obtained from deploying a statistical experimental design. If model (1) is formulated in coded design units then it is assumed that \(z_j\) is a random variable with mean 0 and common variance \(\sigma^2_{z_j}, j = 1, 2, \ldots, r\). Thus, the response surface model for the mean is

\[
E_z(y(x, z)) = f(x) = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i<j}^{k} \beta_{ij} x_i x_j.
\]  

(2)

Model (2) is obtained by taking the expectation of model (1). The response surface model for the process variance is

\[
\sigma^2_{y|z} = V_z(y(x, z)) = \sum_{j=1}^{r} \sigma^2_{z_j} \left( \frac{\partial y(x, z)}{\partial z_j} \right)^2 + \sigma^2
\]

(3)

where \(\sigma^2\) is the residual mean square of the model fit. Model (3) is obtained after applying a conditional variance operator to the first order Taylor series expansion of model (1) [9]. Thus, models (2) and (3) are two separate response surfaces: one for the process mean and one for the process variance. The model for the process variance describes the variability in the response as transmitted from the variability in the hard-to-control variables. Due to the interactions between the controllable and hard-to-control variables the response surface of process variance changes according to changes in the controllable variables. It is therefore possible to determine settings on the controllable variables that reduce or minimize the variability transmitted to the responses.

Montgomery (1999) explained that all the Taguchi engineering objectives for a robust process can now be accomplished through the use of models (1)–(3). For example, the larger-the-better objective can be realized by solving

\[
\text{Max}_x E_z(y(x, z)) \text{ subject to } V_z(y(x, z)) \leq c,
\]

where \(c\) is a given constant representing a maximum value allowable for the process variance. The smaller-the-better objective can be realized by solving

\[
\text{Min}_x E_z(y(x, z)) \text{ subject to } V_z(y(x, z)) \leq c.
\]

To minimize variability around a target the problem

\[
\text{Min } V_z(y(x, z)) \text{ subject to } c_1 \leq E_z(y(x, z)) \leq c_2
\]

can be solved, where \(c_1\) and \(c_2\) are bounds on the process mean. In this paper, robustness studies are aimed at evaluating conditions of the controllable variables that result
in the most sustainable and stable gasifier performance. The approach of response surface methodology as summarized by models (1)–(3) is used in this paper for performing statistical robustness studies.

3 The process and performance variables used for the process robustness studies

The process and performance variables that were selected for the project were discussed in detail in a previous paper [1]. A full $2^3$ factorial design in coal top size, coal bottom size and stone content was utilised, with the inclusion of a centre point. The levels of the process variables are depicted in Table 1. For the purposes of this paper, the experimental levels are given in coded design units [9]. Top and bottom sizes were manipulated by crushing and screening, while stone content was adjusted using dense medium separation (washing). For optimisation purposes a central composite design or another second-order experimental design is preferable [9]. However, since this was a project on a full-scale production plant, the number of tests needed to be minimised. It was previously shown that reliable response surface models could still be constructed on the data [1].

| Experiment no. (Test no.) | Coal top size | Process variables | Stone content |
|---------------------------|---------------|-------------------|---------------|
| 1                         | +1            | −1                | +1            |
| 2                         | 0             | 0                 | 0             |
| 3                         | +1            | +1                | −1            |
| 4                         | +1            | +1                | +1            |
| 5                         | −1            | −1                | −1            |
| 6                         | −1            | +1                | +1            |
| 7                         | +1            | −1                | −1            |
| 8                         | 0             | 0                 | 0             |
| 9                         | −1            | +1                | −1            |
| 10                        | −1            | −1                | +1            |

Table 1: Experimental design layout in coded variables.

The full $2^3$ design gives a total of 8 tests performed with the test gasifier. The centre point was repeated which resulted in a total of 10 tests performed (see Table 1). The inclusion of the centre point enables the modelling of curvature in the data [9]. Additional process variables investigated were the gasifier oxygen load and the CO$_2$ in raw gas concentrations. During each of the 10 tests, loads were varied from low to medium to high, and at each load the CO$_2$ in RG concentration was adjusted between a high and a low value. This gave a total of $10 \times 3 \times 2 = 60$ data points which were used in the statistical evaluation. The statistical evaluation of the data was performed assuming a fully randomised experiment. Due to the number of experiments and the centre point, response surface models of the form in (1) could be constructed on the data. It was also possible to evaluate the significance of quadratic effects of the variables [1]. Thus, due to the experimental design deployed and the ability to construct response surface models, robustness studies could readily be
The PSD of the coal is not uniquely determined by the planned top and bottom sizes. Therefore, three alternative parameters were defined to describe the PSD (see [1] for more details). Three aperture sizes from the American Society for Testing and Materials (ASTM) sieve analysis were selected to define the coal PSD, and for purposes of this paper, will be referred to as the course, medium and fine fractions of the PSD, and were denoted $x_1$, $x_2$ and $x_3$, respectively. Since the three size fractions have to add up to 100% of the coal feed, the three variables describing coal PSD can be regarded as mixture variables [2]. The exact values and ranges of the coarse, medium and fine fractions are of lesser importance to this paper, since the data are only applicable to the particular South African coal that was tested. Optimum size distributions are likely to differ for different coal sources.

A large number of performance variables were considered during the test runs [1]. However, only two of the performance variables which affect sustainable gasifier performance will be discussed in this paper, namely pure gas yield (which is the volume of pure gas produced per mass of dry ash free coal (DAF)), and oxygen consumption per volume of pure gas produced. The actual measured conditions during each test gasifier run deviated slightly from the experimentally planned conditions. Therefore, depending on which variable is classified as the controllable variable, measured process data were used in the statistical evaluations instead of the planned process conditions.

There are two types of variables to be evaluated, i.e. normal process variables and mixture components. Thus, the response surface model in (1) needed to be adjusted to reflect a model in process variables and mixture components [1]. Models (2) and (3) will change depending on which variables are selected to be controllable or hard-to-control. Robustness studies will be performed for the situation where stone is hard-to-control, and PSD is controllable, as well as for the situation where PSD is hard-to-control and stone is controllable. The different approaches to robustness studies and the motivation thereof will be discussed in the subsequent sections.

4 Approaches followed in performing robustness studies

4.1 Approach 1: Stone Content as Hard-To-Control Variable

The variables investigated are $x_1 =$ course fraction, $x_2 =$ medium fraction, $x_3 =$ fine fraction, $w_1 =$ Oxygen load (km$^3$n/h), $w_2 =$ CO$_2$ in RG concentration (vol %) and $z =$ Stone content (mass %). In general, stone content cannot be controlled fully during normal operation without a costly coal beneficiation process, and therefore it is relevant to consider stone content as hard-to-control for the purpose of statistical robustness studies. The other process variables are considered controllable.

As mentioned previously, the actual conditions of the process variables were measured during each test. However, since stone content was one of the design variables in the $2^3$ factorial design, it was natural to use the planned conditions for stone content in the response surface models, and the measured conditions of stone content to define its variability. Montgomery (1999) and Myers and Montgomery (MyeMont1995) also rec-
ommended that known or measured variability of the hard-to-control variables should be used in the modelling of the process variance. The gasifier experimental data were highly suitable for statistical robustness studies, since planned conditions of the process variables were deployed during test runs, but measured conditions were also obtained. Therefore, the planned conditions for stone content were used in the response surface model for the process mean in (2), and the measured conditions were used to define the variability used in the response surface model for the process variance in (3).

Table 2 depicts the ranges observed and subsequently used in the response surface models for the process variables. The variance of stone content was calculated as \( \hat{\sigma}^2_z = (3.132)^2 \) from the measured conditions depicted in Table 2. The variance of stone content was calculated from the data over all the test runs. It is therefore a good representation of the variability of stone content in the run-of-mine coal that might be expected during normal operations. Since the planned conditions for stone content were used in the response surface models, and two levels for stone content were deployed in the factorial design, stone was incorporated into the models as a linear effect only, whereby the interpretation of the robustness analysis was greatly simplified. This is so since the effect of stone is eliminated in the response surface model for the process variance. However, the quadratic effects of the PSD fractions, as well as that of oxygen load and CO\(_2\) in RG concentration, were evaluated. Also, the measured values of the PSD fractions, oxygen load and CO\(_2\) in RG concentration were used in the statistical models. Robustness studies were performed in order to evaluate conditions of oxygen load, CO\(_2\) in RG concentration, and the PSD fractions, that might reduce the effect of the variability or instability of the stone content on sustainable gasifier performance.

### Table 2: Process variables and summary statistics.

| Stone content | Oxygen load | CO\(_2\) in RG | Course fraction | Medium fraction | Fine fraction |
|---------------|-------------|----------------|-----------------|-----------------|---------------|
| planned (mass %) | measured (mass %) |  (km\(^3\)/h) | (vol %) | | |
| Min: 0 | 0.7 | 7.21 | 25.8 | 0.11 | 0.56 | 0.01 |
| Mean: 5 | 4.7 | 9.72 | 26.9 | 0.26 | 0.67 | 0.07 |
| Max: 10 | 9.6 | 12.97 | 28.3 | 0.43 | 0.88 | 0.16 |
| Range: | 10 | 8.9 | 5.77 | 2.6 | 0.33 | 0.33 | 0.15 |
| Total N: 60 | 60 | 60 | 60 | 60 | 60 | 60 |

4.2 Approach 2: PSD Fractions as Hard-to-Control Variables

In general, PSD can be controlled during normal operation. However, conditions of varying PSD can occur under certain circumstances, for example if coal is mined which has a high tendency of break-up during handling and if the coal screening plant is overloaded or experiences operational upsets. Therefore, it is justified to consider the PSD fractions as hard-to-control variables and the other variables, including stone content, as controllable. Robustness studies were performed in order to evaluate conditions of stone content, oxygen load, CO\(_2\) in RG concentration, as well as the PSD fractions that might reduce the effect of variability or instability of the PSD fractions on gasifier performance.

Although top size and bottom size were two of the process variables set at pre-determined
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conditions for the experimental runs according to the $2^3$ factorial design, it was explained earlier that top and bottom size conditions were transformed to three mixture components, $x_1 = $ course fraction, $x_2 = $ medium fraction, $x_3 = $ fine fraction, of the PSD (see also [1]). Furthermore, the measured values of the three PSD fractions were recorded for each test run. For each planned top and bottom size condition, the average of the measured PSD fractions could be calculated. Therefore, the average PSD fractions achieved was considered as the planned PSD fractions for each top by bottom size condition, and used in the response surface model for the process mean in (2). The measured conditions were used to define the variability in the response surface model for the process variance in (3).

Table 2 depicts the ranges observed for stone content, oxygen load and CO$_2$ in RG concentration that were subsequently used in the response surface models for the case where the PSD fractions are the hard-to-control variables. Note that the measured conditions depicted in Table 2 for stone content were now used in the response surface model for the process mean. The variances of the PSD fractions were calculated as $\hat{\sigma}^2_{x_1} = (0.086)^2$, $\hat{\sigma}^2_{x_2} = (0.085)^2$ and $\hat{\sigma}^2_{x_3} = (0.052)^2$ from the measured conditions over all the test runs.

Robustness studies were performed using the response surface models constructed in the process variables. Since the PSD fractions are mixture components it was necessary to evaluate their quadratic effects in the statistical models as well. It was therefore also necessary to evaluate the conditions of the PSD fractions for robustness. Quadratic effects of stone content, oxygen load and CO$_2$ in RG were also evaluated. Note that the factorial experimental design and the nature of the process variables enabled the classification of different types of variables as hard-to-control.

5 Results from the robustness studies

5.1 Evaluation of pure gas yield (m$^3$/ton DAF) under Approach 1

A response surface model was constructed in the process variables for pure gas yield (m$^3$/ton DAF). For the purpose of robustness studies, stone content was incorporated into the model as a linear effect. This was done since stone content was set at two levels and for ease of interpretation. An adjusted $r^2$ value of 0.80 was attained for the model. The response surface model yielded the same trends in the process variables reported previously [1]. This is significant, since the model reported in [1] included a quadratic effect of stone content based on measured operational data. The best yields are obtained at low loads and low stone content for the typical broad PSD and for a CO$_2$ in RG concentration of 26.5%. Also, pure gas yield increases with decreasing top fraction of the PSD for unwashed coal. The model fitting and statistical evaluations were performed on the Design-Expert® software [3].

The main objective of the current statistical evaluations was to evaluate the conditions of the process variables that result in more sustainable pure gas yield production by reducing or eliminating the variability transmitted through the stone content. The final response surface model constructed for pure gas yield of the form in (1) was obtained as

$$ \hat{E}(y(x, w, z)) = 11,180.269x_1 + 808.858x_2 + 193,671.083x_3 + 564.441x_1x_3 $$
where $x_1$ = coarse fraction, $x_2$ = medium fraction, $x_3$ = fine fraction of the PSD, $w_1$ = Oxygen load (km$^3$/n/h), $w_2$ = CO$_2$ in RG concentration (vol %) and $z$ = Stone content (mass %). From (3), the model for the pure gas yield variance is constructed from the final predicted model in (4) and is given by

$$
\hat{\sigma}_y^2 = V \left( \hat{E} \left( y(x, w, z) \right) \right)
= \hat{\sigma}_y^2 \left[ 22.267x_1 + 7.57x_2 + 35.548x_3 - 494.757x_1x_3 ight. \\
\left. -1.655x_1w_1 + 18.930x_1w_2 - 1.753x_2w_1 - 15.550x_3w_1, \right]
\tag{5}
$$

where $\hat{\sigma}_z^2 = (3.132)^2$ is the variance of the stone content calculated from the measured values during all the test runs. Here $\hat{\sigma}_y^2$ is the residual mean square of the statistical model in (4), i.e. $\hat{\sigma}_y^2 = (16.748)^2$. Furthermore $\hat{\sigma}_{y|z}^2$ is the predicted variability in pure gas yield, which includes the variability transmitted from the stone content, as well as the variability from the predicted pure gas yield model. Expression (5) is a function in the various PSD fractions and oxygen load due to the interactions between these variables and stone content (see (4)). Therefore, conditions for PSD and load might be obtained that would result in more sustainable pure gas yield production.

Figure 2 depicts the predicted standard deviation (SD) of pure gas yield production, i.e. $\hat{\sigma}_{y|z} = \sqrt{\hat{\sigma}_{y|z}^2}$ in (5), as a function of oxygen load (km$^3$/n/h) and CO$_2$ in RG concentration (vol %) for various PSD fractions. The smaller the SD of pure gas yield, the more sustainable is pure gas production. It is observed from Figure 2(a) that the smallest variability is transmitted to pure gas yield at middle to high loads for the typical broad PSD. The trend indicates that pure gas yield production is more sustainable at higher oxygen loads.

However, it was shown previously that pure gas yield decreases with increasing oxygen loads [1]. This decrease was more enhanced for unwashed coal. It is now shown here that more sustainable pure gas yield production, although lower, will be achieved at higher loads irrespective of the amount of stone present in the coal. Although the same trends are observed in Figure 2(b) for the finer PSD, i.e. PSD with the coarse fraction reduced, the magnitude of the SD of pure gas yield is much smaller for the finer PSD compared to the typical broad PSD. This result supports previous recommendations to reduce the coarse fraction of the PSD [1].

Various simultaneous objectives can be evaluated through the use of models (4) and (5). Consider the criterion

$$
\hat{E} \left( y(x, w, z = 10) \right) \geq c_1 \text{ subject to } \hat{\sigma}_{y|z} \leq c_2, \tag{6}
$$

where $c_1$ and $c_2$ are given constants. Figure 3(a) depicts the operability region where $\hat{E} \left( y(x, w, z = 10) \right) \geq 1680$ m$^3$/n/t DAF and $\hat{\sigma}_{y|z} \leq 29$m$^3$/n/t DAF simultaneously for the typical broad PSD. In comparison, Figure 3(b) depicts the operability region where $\hat{E} \left( y(x, w, z = 10) \right) \geq 1680$ m$^3$/n/t DAF and $\hat{\sigma}_{y|z} \leq 17$m$^3$/n/t DAF simultaneously for
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Figure 2: Effect of oxygen load (km³n/h) and CO₂ in RG concentration (vol %) on the SD of pure gas yield production (m³n/t DAF) at average stone content (5 mass %): (a) Typical broad PSD, (b) Finer PSD.

The finer PSD, i.e. PSD with the coarse fraction reduced. It is observed, from Figure 3(a), that there is only a small region at middle loads and low CO₂ in RG concentrations where the criteria \( \hat{E}(y(x, w, z = 10)) \geq 1680 \text{ m}^3/\text{t DAF} \) and \( \hat{\sigma}_{y|z} \leq 29 \text{ m}^3/\text{t DAF} \) are satisfied simultaneously for unwashed coal with a typical broad PSD. However, it is observed from Figure 3(b) that there is a large region at higher loads where the criteria \( \hat{E}(y(x, w, z = 10)) \geq 1680 \text{ m}^3/\text{t DAF} \) and \( \hat{\sigma}_{y|z} \leq 17 \text{ m}^3/\text{t DAF} \) are satisfied simultaneously for unwashed coal and the finer PSD. Notice that the SD of pure gas yield in the operability region is much smaller for the finer PSD compared to the typical broad PSD.

Therefore, the statistical robustness studies indicate that the operability region is expanded when the coarse fraction of the PSD is reduced. Furthermore, from Figure 3(b) the SD of pure gas yield production in the operability region is smaller than 17 m³n/t DAF for the finer PSD, which indicates that almost all the variability in production, transmitted from the variability in the stone content, is eliminated. It is therefore concluded that the most sustainable production of high pure gas yield is obtained for a finer PSD, i.e. for a reduction in the coarse fraction of the PSD, and for higher oxygen loads and low CO₂ in RG concentrations. Notice that these conditions provide sustainable production of high pure gas yield irrespective of the amount of stone present in the coal — up to 10% stone which was the maximum value set for the experimental design.

5.2 Evaluation of oxygen to pure gas ratio (m³n/m³n) under Approach 1

To evaluate the effect of the variability in the stone content on the oxygen to pure gas ratio (O₂/PG ratio), which is the volume of oxygen required to produce one volume of pure gas, a response surface model was constructed in the process variables. Again, for the purpose
Figure 3: Operability regions for $\hat{E}(y(x, w, z = 10)) \geq c_1 \text{m}^3\text{n}/\text{t DAF}$ and $\hat{\sigma}_{y|z} \leq c_2 \text{m}^3\text{n}/\text{t DAF}$ simultaneously for unwashed coal: (a) Typical broad PSD with $c_1 = 1680 \text{m}^3\text{n}/\text{t DAF}$ and $c_2 = 29 \text{m}^3\text{n}/\text{t DAF}$, (b) Finer PSD with $c_1 = 1680 \text{m}^3\text{n}/\text{t DAF}$ and $c_2 = 17 \text{m}^3\text{n}/\text{t DAF}$.

of robustness studies, stone content was incorporated into the model as a linear effect. An adjusted $r^2$ value of 0.90 was obtained for the model. The response surface model yielded the same trends in the process variables reported previously [1]. The oxygen consumption per volume of pure gas produced reduces with decreasing loads. Oxygen consumption per volume of pure gas produced increases with increasing top PSD fraction for unwashed coal. Thus, more oxygen is required for coarser coal to produce the same volume of pure gas per ton of DAF coal.

The main objective of the current statistical evaluations was to analyse conditions on the process variables that result in more sustainable consumption of oxygen per volume of pure gas produced, by reducing or eliminating the variability transmitted through the stone content. Firstly, the final response surface model constructed for $O_2$/PG ratio of the form in (1), was

$$
\hat{E}(y(x, w, z)) = 22.6936x_1 - 7.9844x_2 - 54.0801x_3 + 0.3268x_1x_2 \\
+ 0.0827x_1z - 0.0007x_1w_1 - 1.6736x_1w_2 + 16.6053x_2x_3 \\
- 0.0297x_2z + 0.0352x_2w_1 + 0.5881x_2w_2 + 0.1090x_3z \\
- 0.0467x_3w_1 + 3.6876x_3w_2 + 0.0018x_1w_1^2 + 0.0311x_1w_2^2 \\
- 0.0014x_2w_1^2 - 0.0108x_2w_2^2 + 0.0017x_3w_1^2 - 0.0622x_3w_2^2 \\
- 0.0610x_1x_2w_1 - 0.00316x_1zw_2 - 0.1312x_1x_3 \\
- 0.5952x_1x_3w_2 + 0.0012x_2zw_2 - 0.0010x_3zw_2,
$$

where $x_1$ = coarse fraction, $x_2$ = medium fraction, $x_3$ = fine fraction of the PSD, $w_1$ = Oxygen load (km$^3$/h), $w_2$ = CO$_2$ in RG (%) and $z$ = Stone content (%). From (3), the model for the $O_2$/PG ratio variance is constructed from the final predicted model in (7) and is given by

$$
\hat{\sigma}_{y|z}^2 = \hat{V}_z(y(x, w, z)) = V(\hat{E}(y(x, w, z)))
$$
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\[
\hat{\sigma}_z^2 = (3.1319)^2
\]

where \( \hat{\sigma}_z^2 \) is the variance of the stone content calculated from the measured values during all the test runs. Here \( \hat{\sigma}_z^2 \) is the variance of the statistical model in (7), i.e. \( \hat{\sigma}_z^2 = (0.00203)^2 \). Furthermore \( \hat{\sigma}_{y|z}^2 \) is the predicted variability in O\(_2\)/PG ratio, which includes the variability transmitted from the stone content, as well as the variability from the predicted O\(_2\)/PG ratio model. Expression (8) is a function in the various PSD fractions and CO\(_2\) in RG concentration due to the interactions between these variables and stone content (see (7)). Therefore, conditions for PSD and CO\(_2\) in RG concentrations might be obtained that would result in more sustainable oxygen consumption per volume of pure gas produced.

Just from evaluating the coefficients of the variance model in (8) it is found that the variance in the oxygen consumption per volume of pure gas produced will decrease with increasing CO\(_2\) in RG concentrations. Also, the variance in the consumption of oxygen per volume of pure gas produced will decrease if the medium fraction of the PSD is increased. This observation is in line with the general operational philosophy that stable gasifier operation is increased by increasing steam to oxygen ratios, which increases the CO\(_2\) in RG concentration. However, robustness conditions are readily obtained through the evaluation of the simultaneous optimization objective defined in (6). Therefore, Figure 4(a) depicts the operability regions where \( \hat{E}(y(x, w, z = 10)) \leq 0.19 \text{ m}^3\text{n}/\text{m}^3\text{n} \) and \( \hat{\sigma}_{y|z} \leq 0.003 \text{ m}^3\text{n}/\text{m}^3\text{n} \) simultaneously for the typical broad PSD. Figure 4(b) depicts the operability regions where \( \hat{E}(y(x, w, z = 10)) \leq 0.19 \text{ m}^3\text{n}/\text{m}^3\text{n} \) and \( \hat{\sigma}_{y|z} \leq 0.003 \text{ m}^3\text{n}/\text{m}^3\text{n} \) simultaneously for the finer PSD, i.e. PSD with the course fraction reduced.

From Figure 4(a) it is observed that there is only a small region at low loads and high CO\(_2\) in RG concentrations where the criteria \( \hat{E}(y(x, w, z = 10)) \leq 0.19 \text{ m}^3\text{n}/\text{m}^3\text{n} \) and \( \hat{\sigma}_{y|z} \leq 0.003 \text{ m}^3\text{n}/\text{m}^3\text{n} \) are satisfied simultaneously for the typical broad PSD and unwashed coal. However, from Figure 4(b) a large operability region is obtained at middle to high CO\(_2\) in RG concentrations where \( \hat{E}(y(x, w, z = 10)) \leq 0.19 \text{ m}^3\text{n}/\text{m}^3\text{n} \) and \( \hat{\sigma}_{y|z} \leq 0.003 \text{ m}^3\text{n}/\text{m}^3\text{n} \) are satisfied simultaneously for the finer PSD and unwashed coal. Furthermore, all load conditions satisfy these criteria for the finer PSD. The operability region for sustainable oxygen consumption per volume of pure gas produced is significantly expanded for the finer PSD compared to the typical broad PSD. From the robustness studies, it can therefore be concluded that the lowest oxygen consumption per volume of pure gas produced can be achieved at sustainable rates for coal with a reduced top size, middle to high CO\(_2\) in RG concentrations and all load conditions. Notice that these conditions provide sustainable low oxygen consumption per volume of pure gas produced irrespective of the amount of stone in the coal — up to a maximum of 10% stone, as set for the experimental design.

Since both responses, i.e. pure gas yield production and oxygen consumption per volume of pure gas produced, are important in terms of gasifier performance, it is relevant to consider simultaneous optimization of both responses. Consider the criterion

\[
\hat{E}_{PG}(y(x, w, z = 10)) \geq c_1 \text{ m}^3\text{n}/\text{t DAF}
\]

where \( c_1 \) is the criterion for pure gas yield production.
subject to

\[
\hat{E}_\text{PG}(y(x,w,z=10)) \leq c_3 \text{ m}^3/\text{n}^3 \\
\hat{\sigma}_{\text{PG}|z} \leq c_4 \text{ m}^3/\text{n}^3
\]

where \(c_1, c_2, c_3\) and \(c_4\) are given constants, and PG and OPG in the subscripts refer to pure gas yield and O\(_2\)/PG ratio respectively. Figure 5(a) depicts the operability region where the criteria \(\hat{E}_\text{PG}(y(x,w,z=10)) \geq 1620 \text{ m}^3/\text{n}^3 \text{ DAF}, \hat{\sigma}_{\text{PG}|z} \leq 30 \text{ m}^3/\text{n}^3 \text{ DAF}, \)
\(\hat{E}_\text{OPG}(y(x,w,z=10)) \leq 0.19 \text{ m}^3/\text{n}^3 \text{ and } \hat{\sigma}_{\text{OPG}|z} \leq 0.003 \text{ m}^3/\text{n}^3 \text{ are satisfied simultaneously for the typical broad PSD and unwashed coal. In comparison, Figure 5(b) depicts the operability region where the criteria } \hat{E}_\text{PG}(y(x,w,z=10)) \geq 1680 \text{ m}^3/\text{n}^3 \text{ DAF}, \hat{\sigma}_{\text{PG}|z} \leq 17 \text{ m}^3/\text{n}^3 \text{ DAF, } \hat{E}_\text{OPG}(y(x,w,z=10)) \leq 0.19 \text{ m}^3/\text{n}^3 \text{ and } \hat{\sigma}_{\text{OPG}|z} \leq 0.003 \text{ m}^3/\text{n}^3 \text{ are satisfied simultaneously for the finer PSD and unwashed coal.}

From Figure 5(a) it is observed that there is only a small region at medium loads and high CO\(_2\) in RG concentrations where the criteria are satisfied simultaneously for the typical broad PSD and unwashed coal. However, for the finer PSD in Figure 5(b) a much larger region is obtained at middle to high loads and middle to high CO\(_2\) in RG concentrations where the criteria are satisfied simultaneously for unwashed coal. Therefore, considering the robustness of both responses towards the variability in stone content together, the operability region is significantly expanded for the finer PSD compared to the typical broad PSD. The feasible region indicates that a PSD with reduced top fraction and higher middle fraction does provide high sustainable pure gas yield production, as well as sustainable low oxygen consumption per volume of pure gas produced.
5.3 Evaluation of pure gas yield (m$^3$/ton DAF) under Approach 2

An alternative response surface model for pure gas yield production (m$^3$/ton DAF) was constructed in the process variables. This was necessary since the PSD fractions are now considered as the hard-to-control variables. The other process variables, including stone content, are considered as controllable. Therefore, for the purpose of the statistical evaluations, the PSD fractions were defined as the average PSD achieved for each top by bottom size condition prepared. This was then considered as the planned PSD fractions. For the purpose of robustness studies the SD for each PSD fraction was calculated from the measured data observed during all the test runs. Therefore, the PSD fractions defined as such constitutes a mixture with each component in the mixture having a SD for robustness studies. Also, for this approach, since stone content is now considered as controllable, the measured stone content at each test period was used for the statistical evaluations. Therefore, the quadratic effect of stone content was incorporated into the statistical model.

An adjusted $r^2$ value of 0.82 was obtained for the model. It was also found that the response surface model, although slightly different in structure compared to the model for Approach 1, yielded the same trends in the process variables reported previously [1]. The main objective of this approach was to evaluate the conditions of the process variables that result in more sustainable pure gas yield production by reducing or eliminating the variability transmitted through the PSD fractions. The model for the pure gas yield variance is constructed from the final predicted model and is given by

$$\hat{\sigma}_{y|x}^2 = \hat{V}_x(y(x, w, z)) = V(\hat{E}(y(x, w, z)))$$

$$= \hat{\sigma}_{x_1}^2 [17256.482 - 2070.401z_3 + 181.260z - 1848.216w_1 - 480.091w_2$$

$$+ 7.975z^2 + 26.383w_1^2 - 3.975zw_1 - 9.069zw_2 + 49.660w_1w_2]^2$$

**Figure 5:** Operability regions for $\hat{E}_{PG}(y(x, w, z = 10)) \geq c_1$ m$^3$/t DAF, $\hat{\sigma}_{PG|z} \leq c_2$ m$^3$/t DAF, $\hat{E}_{OPG}(y(x, x, z = 10)) \leq c_3$ m$^3$/m$^3$ and $\hat{\sigma}_{OPG|z} \leq c_4$ m$^3$/m$^3$ simultaneously: (a) Typical broad PSD with $c_1 = 1620$, $c_2 = 30$, $c_3 = 0.19$ and $c_4 = 0.003$, (b) Finer PSD with $c_1 = 1680$, $c_2 = 17$, $c_3 = 0.19$ and $c_4 = 0.003$. 
\[
+\sigma^2_x [11781.596 + 20.271z + 614.713w_1 - 944.333w_2 - 10.581w_1^2] \\
+19.721w_2^2 - 2.388zw_1 - 14.938w_1w_2]^2 \\
+\sigma^2_x [2467.624 - 2070.401x_1 + 113.671z - 82.189w_1 - 30.443z^2] \\
+20.022zw_1]^2 + \hat{\sigma}^2,
\]

where \(x_1\) = course fraction, \(x_2\) = medium fraction, \(x_3\) = fine fraction of the PSD, \(w_1\) = Oxygen load (km\(^3\)n/h), \(w_2\) = CO\(_2\) in RG concentration (vol \%) and \(z\) = Stone content (mass \%). Recall that \(\hat{\sigma}^2_x = (0.086)^2\), \(\hat{\sigma}^2_{x_2} = (0.085)^2\) and \(\hat{\sigma}^2_{x_3} = (0.052)^2\) are the variances for the coarse fraction, medium fraction and the fine fraction, respectively. These variances were calculated from the actual measured fractions during all the test runs. Here \(\hat{\sigma}^2 = (15.836)^2\) is the variance of the response surface model. Notice that the model variance obtained for this approach is slightly smaller compared to the model for Approach 1. This approach therefore produced an improved model. Furthermore \(\hat{\sigma}^2_{y|x}\) is the variance of pure gas yield, which includes the variability transmitted from the PSD fractions, as well as the variability from the predicted pure gas yield model.

Firstly, note that model (10) include the coarse fraction and the fine fraction of the PSD as parameters. The PSD fractions were not eliminated in the variance model due to the interactions between the various PSD fractions present in the response surface model. Therefore, it is desirable to seek conditions not only on the process variables stone content, load and CO\(_2\) in RG concentrations, but also on the PSD fractions, that result in sustainable pure gas yield production. However, it is also noticeable that the complexity of model (10) is much higher than that of model (5) obtained under Approach 1. Therefore, the interpretation of the variability in pure gas yield as transmitted from the variability in the PSD fractions will be much more difficult. However, it was possible to generate contour plots with the Design-Expert\textsuperscript{\textregistered} software that highlight the important trends towards sustainable production. The reader is advised to consult the Design-Expert\textsuperscript{\textregistered} User’s Guide [3] for an explanation of the variance model in the dual response surface approach for mixture components.

Figure 6 depicts the predicted standard deviation (SD) of pure gas yield production, i.e. \(\hat{\sigma}_{y|x} = \sqrt{\hat{\sigma}^2_{y|x}}\) in (10), as a function of PSD fractions for different CO\(_2\) in RG concentrations and medium loads. It is observed from Figure 6(a) that the smallest variability is transmitted to pure gas yield at high medium fraction for medium loads and low CO\(_2\) in RG concentrations for unwashed coal. The trend indicates that the sustainability of pure gas yield production improves as the medium fraction increases. The same trend is observed in Figure 6(b) that depicts the variability of pure gas yield for medium loads and high CO\(_2\) in RG concentrations for unwashed coal. However, the decrease in the SD of pure gas yield, or the improvement in sustainability, is more enhanced for lower CO\(_2\) in RG concentrations, as depicted in Figure 6(a). Therefore, as observed previously, more sustainable pure gas yield production will be achieved by reducing the coarse fraction and increasing the medium fraction of the PSD for unwashed coal. These observations indicate that the statistical robustness studies were performed correctly and that it was applicable to this data set.
5.4 Evaluation of oxygen to pure gas ratio (m$^3$n/m$^3$n) under Approach 2

To evaluate the effect of the variability in the PSD fractions on the oxygen to pure gas ratio ($O_2$/PG ratio), which is the oxygen consumption per volume of pure gas produced; a response surface model was constructed in the process variables. An alternative model for $O_2$/PG ratio was constructed for this approach, due to the fact that the PSD fractions are now considered as the hard-to-control variables. The adjusted $r^2$ value of the response surface model with this approach was founded to be 0.89. It was also found that the response surface model, although slightly different in structure compared to the model for Approach 1, yielded the same trends in the process variables as was reported earlier [1].

The main objective of this approach was to evaluate the conditions of the process variables that result in more sustainable consumption of oxygen per volume of pure gas produced by reducing or eliminating the variability transmitted through the PSD fractions. The model for the $O_2$/PG ratio variance is constructed from the final predicted model and is given by

$$\hat{\sigma}_y^2 = \hat{V}_x(y(x, w, z)) = V(\hat{E}(y(x, w, z)))$$

$$= \hat{\sigma}_{x1}^2(24.7480 - 0.2951x_3 + 0.1734z + 0.0389w_1 - 1.8776w_2$$

$$+ 0.0016z^2 - 0.0017w_2^2 + 0.0356w_2^2 - 0.0069zw_1^2)$$

$$+ \hat{\sigma}_x^2 - 13.8555 - 0.0936z - 0.0021w_1 + 1.0621w_2 - 0.0014z^2$$

$$+ 0.0002w_1^2 - 0.0201w_2^2 + 0.0039zw_1^2$$

$$+ \hat{\sigma}_{x3}^2[15.7458 - 0.2951x_1 + 0.0359z - 0.0018w_1 - 1.1643w_2$$

$$+ 0.0089z^2 + 0.0002w_2^2 + 0.0220w_2^2 - 0.0043zw_1^2] + \hat{\sigma}^2$$

where $x_1$ = coarse fraction, $x_2$ = medium fraction, $x_3$ = fine fraction of the PSD, $w_1$ = Oxygen load (km$^3$n/h), $w_2$ = CO$_2$ in RG concentration (vol %) and $z$ = Stone content (mass %). Here $\hat{\sigma}^2 = (0.0022)^2$ is the variance of the response surface model. The precision of the model is very similar to the model obtained under Approach 1.

Model (11) includes the coarse fraction and the fine fraction of the PSD. The PSD fractions were not eliminated in the variance model, due to the interactions between the various PSD fractions present in the response surface model. Therefore, it is desirable to seek conditions not only on the process variables stone content, oxygen load and CO$_2$ in RG concentrations, but also on the PSD fractions, that result in sustainable consumption of oxygen per volume of pure gas produced. However, since both responses, i.e. pure gas yield production and oxygen consumption per volume of pure gas produced, are important in terms of gasifier performance, it is relevant to consider simultaneous optimization of both responses. Consider the simultaneous criteria in (9).

Figure 7(a) depicts the operability regions where the criteria $\hat{E}_{PG}(y(x, w, z = 10)) \geq 1650$ m$^3$n/t DAF, $\hat{\sigma}_{PG|z} \leq 35$ m$^3$n/t DAF, $\hat{E}_{OPG}(y(x, w, z = 10)) \leq 0.21$ m$^3$n/m$^3$n and $\hat{\sigma}_{OPG|z} \leq 0.008$ m$^3$n/m$^3$n are satisfied simultaneously for the typical broad PSD and unwashed coal. In comparison, Figure 7(b) depicts the operability regions where the criteria $\hat{E}_{PG}(y(x, w, z = 10)) \geq 1650$ m$^3$n/t DAF, $\hat{\sigma}_{PG|z} \leq 31$ m$^3$n/t DAF, $\hat{E}_{OPG}(y(x, w, z = 10)) \leq 0.21$ m$^3$n/m$^3$n and $\hat{\sigma}_{OPG|z} \leq 0.008$ m$^3$n/m$^3$n are satisfied simultaneously for the finer PSD and unwashed coal. The small operability region obtained for the typical broad PSD in Figure 7(a) supports earlier results that the typical broad PSD does not provide.
Figure 6: Effect of PSD fractions on the SD of pure gas yield production (m$^3$/n/t DAF) for unwashed coal: (a) Medium oxygen load and low CO$_2$ in RG concentration, (b) Medium oxygen load and high CO$_2$ in RG concentration.

Another very important result is obtained through the robustness studies by comparing the case of PSD fractions as hard-to-control variables to the case where stone content is the hard-to-control variable. For the case where stone content is the hard-to-control variable the operability region is significantly expanded towards higher load conditions for the finer PSD. Higher loads cannot be obtained if PSD fractions cannot be controlled. This is a very important result, since higher loads is a strategic objective for the gasification plant in the future. It is therefore more important and also more efficient to control PSD fractions rather than to control stone content. If the PSD is controlled then higher loads can be achieved. Therefore, the robustness studies have shown that reducing the top size of the PSD, as well as controlling the PSD fractions during normal operation, yields sustainable high pure gas yield production and low oxygen consumption per volume of pure gas produced at sustainable rates.
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6 Conclusions

Through the application of statistical robustness studies the objectives in experimentation mentioned by Montgomery (1999) have been addressed and solved, i.e. to determine where to set the influential controllable variables so that the variability in the responses are small, and to determine where to set the influential controllable variables so that the effects of the hard-to-control variables on the response are small. In addressing this problem two approaches of robustness studies were followed, i.e. considering stone content as the hard-to-control variable, and PSD as controllable, as well as considering the PSD fractions as hard-to-control variables, and stone content as controllable. The classification of the different types of variables as hard-to-control variables could only be performed due to the deployment of the statistical experimental design.

Previously, it was shown that pure gas yield production is maximised at low oxygen loads and maximum medium fraction of the PSD for unwashed coal [1]. However, as mentioned earlier, a beneficiation plant would add considerable cost to the gasification process. In this paper, it was shown that sustainable high pure gas yield production, as well as lower oxygen consumption per volume of pure gas produced, can be achieved by reducing the coarse fraction of the PSD fed to the gasifier. The medium fraction of the PSD needs to be maximized to ensure that the variability in the responses is small. For stone as the hard-to-control variable, and PSD fractions as controllable, this improvement in sustainability and throughput can be achieved irrespective of the amount of stone present in the coal — up to 10% stone, which was the maximum value set for the experimental design. However, for PSD fractions as the hard-to-control variables, and stone content as controllable, maximum sustainability and throughput is also achieved by reducing the coarse fraction of the PSD for unwashed coal. Another important result is that higher oxygen loads cannot be achieved if PSD fractions are hard-to-control. For stone content as
hard-to-control, and PSD as controllable, the operability region for improved sustainability and throughput is significantly expanded towards higher loads.

Although optimum process conditions for maximum throughput were previously reported [1], the optimum conditions for maximum throughput and sustainability of gasifier operability were not known prior to this study. The importance to control the PSD, rather than the stone content, was also not common knowledge previously. Through the application of statistical robustness studies significant contributions could be made to the understanding of gasification and to the strategic growth of the gas production business. This study emphasizes the importance of statistical experimental design and response surface modelling in process and product improvement and optimization. The methods and results presented in this paper provide practical evidence that the dual response surface approach is a powerful method for performing robustness studies.

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