UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

DEVELOPMENT OF A DEVICE FOR MEASUREMENT AND CONTROL OF TOP PRODUCT COMPOSITION IN A DISTILLATION COLUMN

by

KALYANA SRINIVASA CHAKRAVARTHY NANDURI
(Srinu)

A dissertation submitted in partial fulfilment
of the requirements for the degree

MASTER OF ENGINEERING (Control Engineering)

Department of Chemical Engineering

Faculty of Engineering

UNIVERSITY OF PRETORIA

OCTOBER 1999

© University of Pretoria

TABLE OF CONTENTS

Chapter FIVE : Discussion of Results

Appendices I-III

List of References

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

TITLE: **Development of a device for measurement and control of top product composition in a distillation column**

SUPERVISOR: Prof P L de Vaal

DEPARTMENT: Chemical Engineering

UNIVERSITY: University of Pretoria

DEGREE: Master of Engineering
(Control Engineering)

SYNOPSIS

The objectives of this investigation were to :

Develop a capacitance measuring device that can be used for measuring composition of ethanol-water mixtures.

Implement this measurement technique on a laboratory distillation column for top product composition measurement and control.

Compare the results obtained in this manner with previously used inferred measurements by temperature.

Experiments were conducted to calibrate the capacitance cell ie, to obtain a relation between composition of an ethanol and water mixture and the output of the capacitance cell. Open loop tests were executed on the distillation column to determine if the capacitance device had any negative influence on measurement compared to composition measurements via temperature. Top product composition control using the capacitance cell under closed loop conditions was also tested, to observe the capacitance cell's performance.

The capacitance cell gave repeatable readings during the calibration procedure. In the open loop tests, process models were obtained for control purposes and no negative influence was observed. The capacitance cell's closed loop responses were observed to be satisfactory. It was simple to construct, easy to operate and proved to be a relatively inexpensive device.

In the final analysis, the capacitance cell proved to be a useful device in direct measurements of composition of binary mixtures, and offers scope for further development in its application to the control of top product composition in distillation columns of industrial scale.

KEY WORDS: Distillation column control, composition measurement, capacitance.

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

TITEL: **Ontwikkeling van ´n instrument vir meting en beheer van topproduksamestelling in ´n distillasiekolom**

LEIER: Prof P L de Vaal

DEPARTEMENT: Chemiese Ingenieurswese

UNIVERSITEIT: Universiteit van Pretoria

GRAAD: Magister in Ingenieurswese
(Beheeringenieurswese)

SINOPSIS

Die doelwitte van hierdie ondersoek was om:

- 'n kapasitansiegebaseerde meetinstrument te ontwikkel wat gebruik kan word om samestelling van etanol-watermengsels te meet.
- hierdie meettegniek te implementeer op 'n laboratoriumskaal distillasiekolom vir meet en beheer van topproduksamestelling.
- die resultate op hierdie wyse verkry te vergelyk met metings wat deur middel van temperatuurmetings afgelei is.

Eksperimente is uitgevoer om die kapasitansiesel te kalibreer, dws om 'n verband tussen die samestelling van 'n etanol-watermengsel en die uitset daarvan te verkry. Ooplus traptoetse is op die distillasiekolom uitgevoer om vas te stel of die kapasitansiesel enige nadelige invloed het op die dinamika van die samestellingmetingsproses in vergelyking met samestellingbepaling deur middel van temperatuurmeting. Die werkverrigting van die kapasitansiesel onder geslotelustoestande is ook getoets.

Die kapasitansiesel het herhaalbare lesings gelewer tydens die kalibrasieproses. Tydens die ooplustoetse is prosesmodelle verkry wat vir beheerderontwerpdoeleindes gebruik kan word Geen nadelige newe-effek as gevolg van die gebruik van die kapasitansiesel in die meetlus kon waargeneem word nie. Die geslotelusrespons van die sisteem met die kapasitansiesel was ook bevredigend. Die sel is relatief goedkoop, eenvoudig om te vervaardig en maklik om te bedryf. Opsommenderwys kan dit gestel word dat die kapasitansiesel 'n besonder bruikbare instrument is vir die meting van konsentrasie in binêre mengsels. Dit hou moontlikhede in vir verdere ontwikkeling as deel van die meet en beheer van topproduksamestelling op industriële distillasiekolomme.

SLEUTELWOORDE: Distillasiekolombeheer, bepaling van samestelling, kapasitansie.

Nomenclature

Symbol	Description	Units
A	Antoine constant	
A.C	Alternating current	[A or mA]
A/D	Analog to Digital	
A	Composition determined via measurement technique	[% mass]
a	Inner diameter of a cylindrical capacitor	[cm or m]
a	a constant(temperature relations)	
B	Antoine constant	
B	Second Virial coefficients	
B	Composition determined via reference testing method	[% mass]
b	Outer diameter of a cylindrical capacitor	[cm or m]
C	Capacitance	[F or mF]
C	Antoine constant	
c	constant(fugacity & pressure relation)	
C	Composition	[% mass]
D/A	Digital to analog	
D.C	Direct current	[A or mA]
d	Distance between plates of a parallel plate capacitor	[cm or m]
f	Fugacity	[pa or mm Hg]
$\hat{f}_i$	Fugacity of component i in solution	[pa or mm Hg]

Symbol	**Description**	**Units**
f_i^0	Fugacity of component i in standard state(STP).	[pa or mm Hg]
f	Frequency	[Hz]
F	Volumetric flow rate of a liquid	[m^3 /sec]
I/O	Input-output	
K	Gain	
L	Length of a cylindrical capacitor	[cm or m]
L	% change in the reflux valve position	
m	An input	
mV	Millivolts	
P	Pressure	[pa or mm Hg]
Q	Heat supplied by the thyristor	[cal or kcal]
R	Gas constant	
S	Surface area of a parallel plate capacitor	[cm^2 or m^2]
T	Temperature	[K or ^0C]
T	Time	[sec or hrs]
V	Volume of space in a capacitor	[cm^3 or m^3]
V	Volume of individual compounds	[cm^3 or m^3]
V	Voltage/Volts	[mV or V]
x	Mole-fraction of a component in liquid phase (ideal)	
x^1	Mole-fraction of a component in liquid phase (non-ideal)	
x	Filtered values	

Symbol	**Description**	**Units**
y	Mole-fraction of a component in gas phase (ideal)	
y^1	Mole-fraction of a component in gas phase (non-ideal)	
y	Output of a process, also unfiltered output	
1	Ethanol	
2	Water	

Characters

γ	Activity coefficient
ω	Acentric factor
ϵ	Permittivity
Φ	Fugacity coefficient
τ	Time
α,β	Van-Laar constants
μ	Mean
σ	Standard deviation

Subscript

c	Controller coefficient
cy	Cylindrical capacitor
pa	Parallel plate capacitor
I	Integral
i	Component i
i	Pure liquid i
j	Pure liquid j

eth	Ethanol
wat	Water
n	Sampling interval
p	Process
p-p	peak to peak
d	Dead (as in $\tau_{d,}$ ie, dead time)
r	Output
r	Reduced(as in reduced P or T)
s	Input
L	% change in reflux valve position
Q	Heat added to the reboiler
1	Ethanol
2	Water

Superscript

sat	Saturated
0	Related to Virial coefficient
1	Related to Virial coefficient

List of Tables

Page no.

Appendix Tables

List of Figures

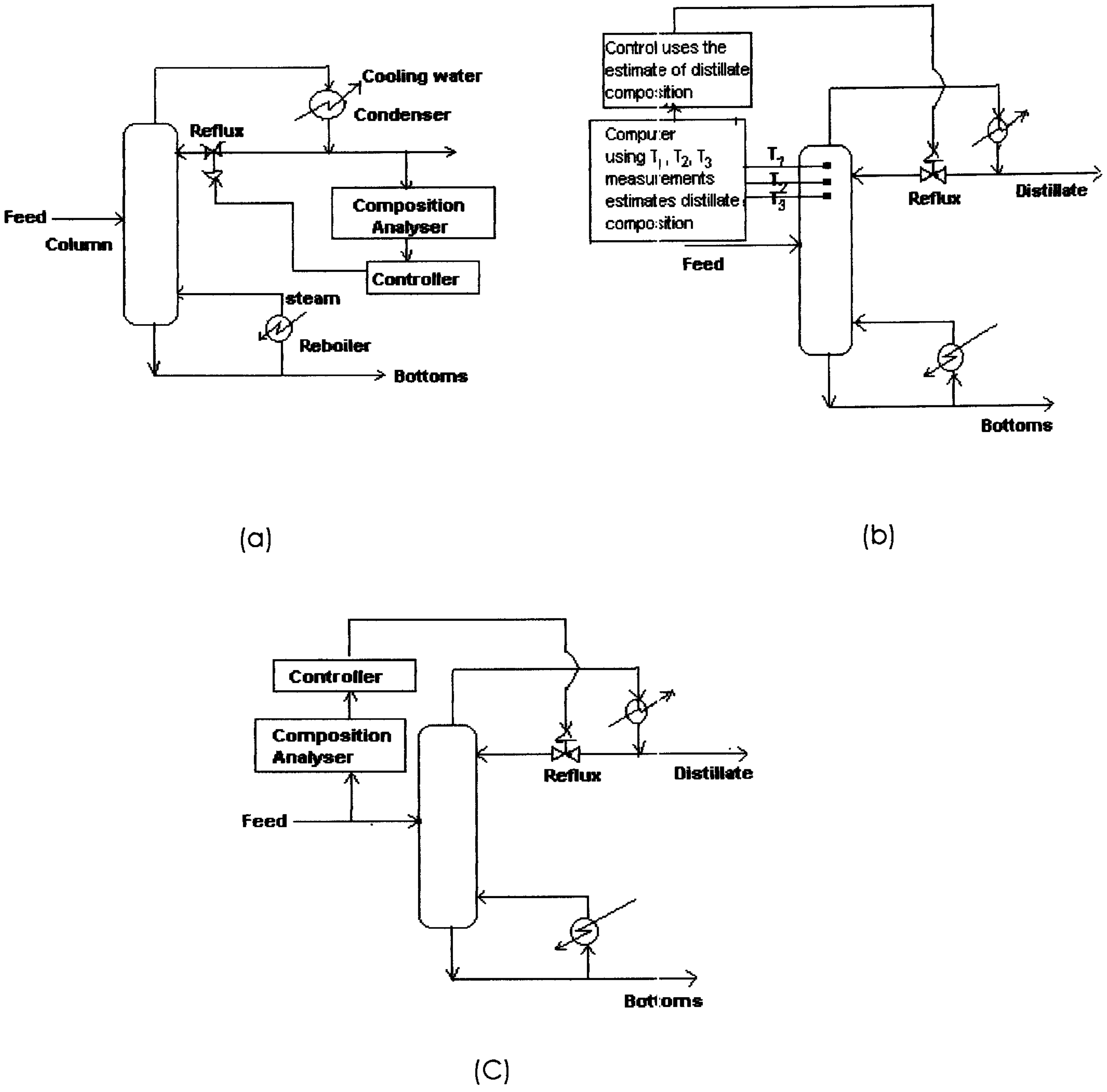
Page no.

Chapter ONE

Introduction

In distillation column control, accurate and reliable measurement of top product composition is a primary requirement. There are various measurement techniques in use which are classified as (a) direct and (b) inferred ,depending upon the type of measurement. Typical examples of direct measurement techniques are chromatographs and various types of spectroscopic analysers. They are used to measure composition in terms of one or two key components or in terms of all the components present in the process stream. The dominant feature of chromatographic analyzers is the dead time which can be quite large. The time required for the sample to travel from the process stream to the column , plus the time required through the column plus the time required by the detector at the end of the column to respond, can be quite large. Such large dead times can result in ineffective control. Other features of the composition analyzers stated above are 1) their low operational reliability and 2) relatively high cost. Inferred measurements on the other hand, use temperature to infer composition and rely heavily on the accuracy of the estimator for composition measurement. Thus it is understood that in practice, composition measurement techniques have several disadvantages (Stephanopolous, 1984).

In the next step in column control, the measurement techniques are applied in an appropriate control configuration, namely direct feedback, inferential feedback or feedforward control, as shown below:



(a) (b)

(C)

Figure 1.1 Common configurations for distillate composition control of a simple distillation column: (a) Feedback; (b) Inferential (c) Feedforward; (Stephanopolous, 1984)

The objective of the three control configurations mentioned above is to keep the controlled variable, in this case composition , at the desired value. Feedback control configurations, as in Figure 1.1 (a) use direct measurements of the composition to adjust the value of the manipulated variable, the reflux flow rate. Inferential feedback control uses secondary measurements of compositions using temperature to adjust the value of the reflux flow rate. In order to calculate composition the estimator in Figure 1.1 (b)uses the values of the measured outputs, together with thermodynamic relations . Feedforward control, as in Figure 1.1 (c) uses direct measurement of disturbance, in this case the feed composition, to adjust the value of the manipulated variable(reflux flow rate), based on an available process model. From the above discussion, it follows that a direct, effective and cheaper method of composition measurement that gives smaller dead times, more accurate measurements and a device which can easily be applied to the above cited control configurations in a distillation column is highly desirable. In this study it is shown that a device that uses the principle of variation in capacitance as a means of measuring composition in a binary mixture can be successfully applied. This will facilitate computerized on-line measurement and control of composition in a distillation column and can also be integrated in plant wide control.

The aim of this project is therefore :

(i) To study the basic characteristics of a capacitance cell as applied to direct measurement of composition of binary mixtures.

(ii) To provide details of the development and testing of a capacitance cell.

iii) To use this cell for controlling the top product composition of a laboratory scale distillation column, to compare these results with the temperature based composition controller used previously and to establish whether or not the results obtained show an improvement over the other methods in use.

This dissertation consists of the following chapters :

A literature survey, in which the relation between composition and dielectric constant of liquid mixtures as well as the theory of capacitors and different types of capacitors used will be presented, specifically A.C and D.C circuit theories in the context of choice of application of the former in the device. A description of the chromatographic methods and infra - red analysers, looking at their disadvantages, followed by temperature-composition correlations and their negative effects is also given.

In the chapter on process equipment a description of the capacitance cell and its associated electronics , the laboratory scale distillation column and data- converter cards is given.

Test results obtained on the column form the contents of the following chapter which contains the results of the calibration process and provides the experimentally derived relation between composition and capacitance. Temperature and capacitance measurements are used to obtain dynamic models for the distillation column using step tests. PI controllers are used to provide control action for the top product composition, using a thyristor and the reflux valve as the final control elements.

In the subsequent chapter results will be examined, interpreted and critically discussed. In the last chapter conclusions and recommendations are presented.

Chapter TWO

Literature Survey

Introduction:

In this chapter a detailed survey of the composition measurement techniques, both direct and inferred which are presently in use such as chromatographic, infra-red analysers, refractive index and density, and temperature methods along with their disadvantages and limitations will be presented. This will be followed by the theory and principles of the proposed capacitance cell and its application to the measurement of composition of ethanol and water mixtures.

This chapter consists of the following topics:

1)Chromatographic methods for determining composition.

2)Applications of infra-red analysers for chemical analysis.

3) Refractive index and density methods for composition determination.

4) Thermodynamic relationships between composition and temperature.

5) Evaluating ethanol and water composition using non-ideal relations.

6) Disadvantages of using temperature measurement as a means of inferring composition measurement and control.

7) A relation between composition and dielectric constants of ethanol-water mixtures.

8) The theory of capacitors, looking in detail at the advantages of using a parallel plate capacitor over a cylindrical capacitor.

9)The significance of using an alternating current (A.C) circuit instead of a direct current (D.C) circuit in the measuring device.

2.1 Chromatographic techniques used in distillation column control

Gas chromatography(Schomburg,1990), is a direct measurement technique used in determining gas or liquid composition.

Their relatively limited applications in direct measurement of composition in a distillation column is because of their large dead times, and high costs under typical operating conditions . The problem of their dead times is due to liquid components to be analysed, requiring different times of analysis. This is explained by the principle of operation which is shown in Figure 2.1 below:

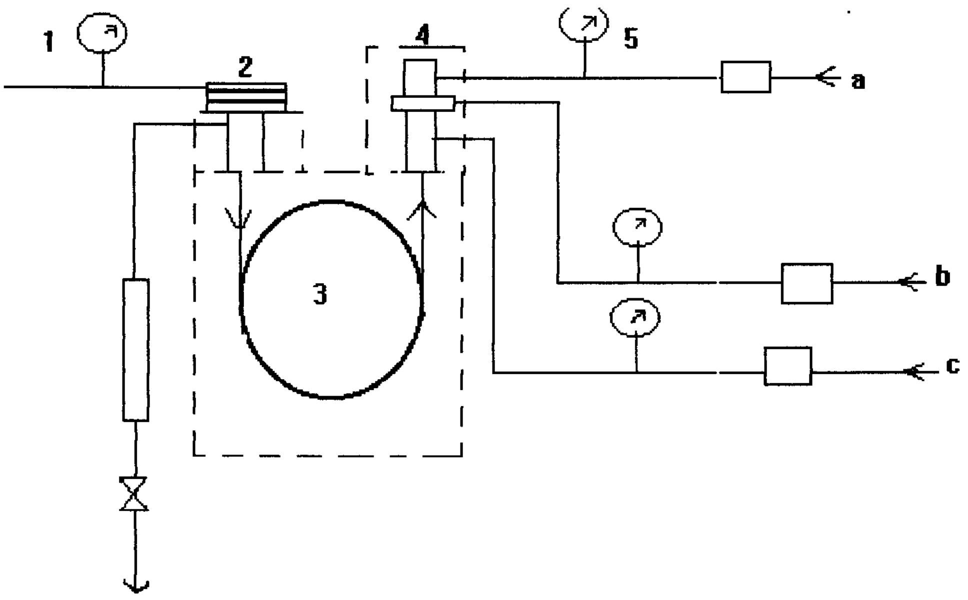


Figure 2.1 Elementary parts of a Gas Chromatograph(Schomburg,1990)

1	carrier gas supply
2	injector for split or splitless sample introduction
3	column (packed, capillary)
4	detector
5	gas supply for Flame ionization detector (FID), (a) air, (b) hydrogen, (c) make-up gas (argon or nitrogen)

The two elementary parts of a gas chromatographic system are the column (3), in which the separation takes place, and the detector (4). The carrier gas supply unit (1), has as main component, a pressure and flow regulator connected to a gas cylinder, containing the inert carrier gas (H_2, He , N_2 or Ar). The sampling device, or injector (2), introduces the sample to be analysed into the carrier gas against an inlet pressure of the inert gas which may be under pressures between 0,01 and 0,5 Mpa depending on the type of the carrier gas and the flow resistance. The injector is usually heated and operated at a temperature which permits the volatilization of liquid samples.

The sample components are separated by dilution and mixing with the carrier gas flow, this separation being facilitated by the fact that the column is situated in an oven compartment. The temperature of the oven is changed linearly to provide the temperature necessary for the optimum separation of the components to be analysed.

The efficiency of the column is determined by selectivity of the stationary phase present in the column and the sample capacity, ie the amount of sample that can be separated. The sample capacity depends upon the amount of stationary phase contained in the column.

The separated sample components enter the detector with a characteristic profile of their concentrations within the carrier gas. The registration system produces continuously recorded , time dependent electrical signals. Detectors that are generally used in gas chromatography are flame ionization detectors (FID) and thermal conductivity detectors(TCD).

The time differences in analysis of the sample components cause major transportation lags(dead times) in the control loop. This will result in degrading performance of the controller. Due to their delicate nature , they generally require careful handling ; they are therefore mounted on the ground for ease of maintenance.(Poole.S.K & Poole .C.F, 1991)

2.2 *Infra-red spectroscopy*

This method of analysis, (Meehan, 1966), (Pecksok & Shields, 1968), (Olsen,1975) is based on the measurement of the interaction between matter and electromagnetic radiation in the region of 1-25 µm. The intensity of radiation energy absorbed or emitted due to transfer of electrons to and from the characteristic energy level is measured . The absorption/ transmission pattern is unique for any chemical compound.

The radiation source used in infra-red spectrophotometers is one of the following:

(i) The Nernst Bar: a hollow zirconium or yttrium oxide rod electrically heated to 1500 °C.

(ii) Globar : A silicone carbide bar heatable to 1200 °C.

Detectors used in infra-red analysers are :

In the near infra-red region (0.8 -3 µm) the photo conductive cell is used. Upon illumination with radiation of appropriate wavelength the electrons are raised to the conducting bands.

(ii) In the middle and far infra-red regions the infra red (IR) photons are absorbed and their energies are converted to thermal energy. Rapid response thermometers are therefore used as detection devices

Examples are :

Thermocouples (gold -tellurium)
Resistance bulb thermometers
Gas thermometers

In IR devices the sample is placed before the monochromator so that the monochromator can remove unnecessary radiation emanating from the sample and cell before the detector is reached.

The major problem experienced during application of a infrared analyser is that it uses a narrow cell in which the sample is placed. Calculating composition therefore becomes difficult, since indices (absorption and extinction coefficients)used in composition determination of the analysers require an accurate measurement of sample thickness. Infra-red analysers also use wide slit widths, the slits which form part of infra-red spectrometers, provide a better spectral resolution and accuracy depending on their width and are close to 900 nM. This can cause problems because the energy available in the useful wavelength range is relatively small and a very slight shift in the wavelength would cause a considerable change in the indices, creating a significant error in composition.

In summary, spectroscopic analysers such as infrared analysers are to a large extent accurate, but expensive and require careful calibration and maintenance.

2.3 *Refractive Index (R.I)Methods and Density methods.*

The relation between refractive index and the composition range under consideration (75-90%) was obtained from the International Critical Tables (ICT,1930)and is shown in Figure 2.2. From the figure, a curvature which implies that a single measurement indicates two different values of composition is observed. Furthermore the refractometer that is available in the laboratory, reads values of refractive index only up to the third decimal value. Therefore it is only possible to determine composition to the nearest 0,001 R.I units, which is clearly not accurate enough. Refractive index methods were therefore found to be disadvantageous.

The density method, on the other hand, provides a relation between density of the mixture and composition as shown in Figure 2.3(Perry,1987).It is reliable, avoiding difficulties noticed by refractive index measurements.

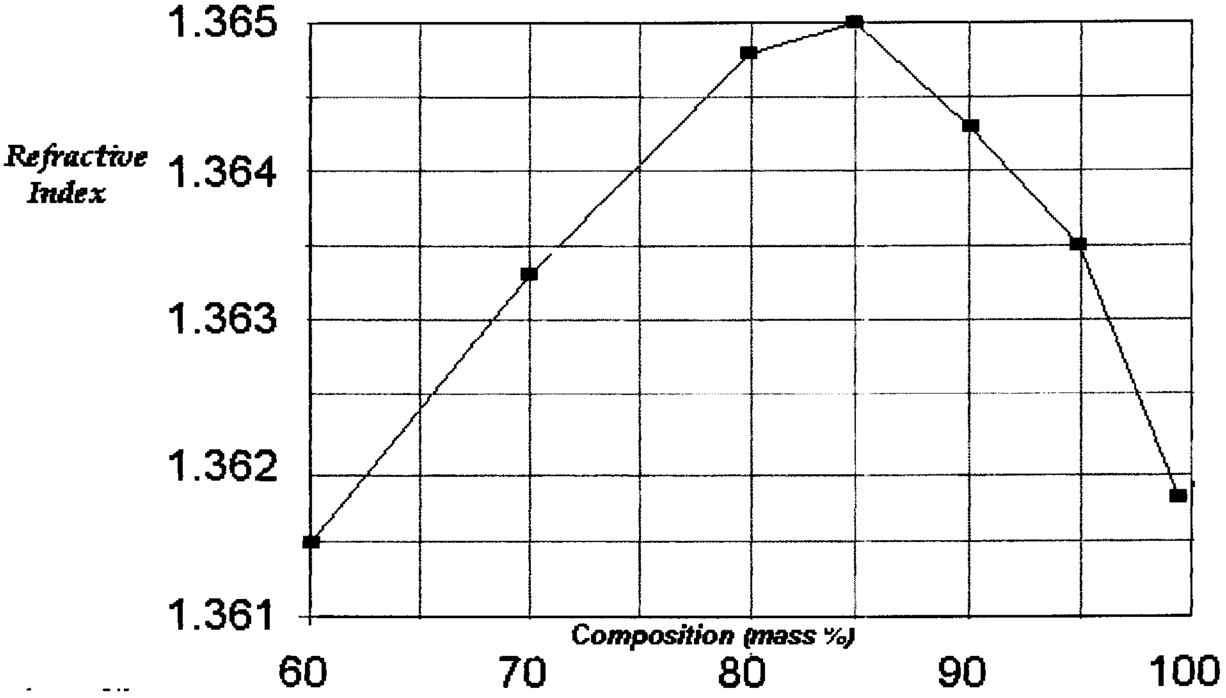


Figure 2.2 A relation between composition and refractive index of ethanol and water mixture at 20 ^{0}C.(International Critical Tables, 1930)

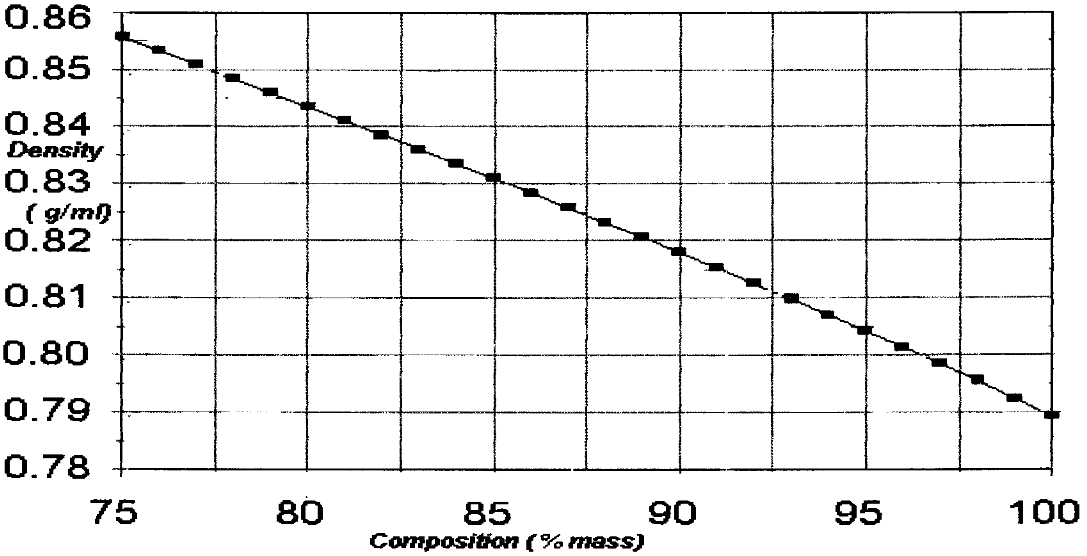


Figure 2.3 A relation between composition and density of ethanol and water mixture at 20 ^{0}C.(Perry , 1987)

The accuracy with which density of a sample can be determined using gravimetric analysis is ±0,00001 g/mL(Manahan, 1986). Although this method is useful in determining composition, density measuring methods commercially available are very large in size and expensive and difficult to install and operate as a measurement technique in a small scale distillation column(Svrceck,1967).

2.4 *Temperature -Composition Correlations (Ideal)*

Temperature can be used as an inferred measurement to evaluate the composition of binary mixtures like ethanol and water in this case, assuming ideal conditions. In distillation columns, top product composition is inferred by placing a thermocouple in the top tray, because not only will it better reflect the condition of the distillate but also temperature profiles at the ends of a column tend to become flat where the products reach purity(Shinskey, 1988). The composition inferred by temperature is calculated using equations which relate the partial pressure of a component of a mixture to its composition (in mole fraction) in the vapour phase as derived from Dalton's law and the ideal gas law (Hougen, Watson & Ragatz,1976), Raoult's law which relates the partial pressure of a component of a mixture to its composition (in mole fraction) in the liquid phase, and the Antoine equation which relates the vapour pressure of an individual component to the prevailing temperature, given by Equation 2.2. (Smith & Van Ness, 1987)

$P_i = x_i P_i^{sat}$ Raoult's law

$P_i = y_i P$ Derived from Dalton's and the ideal gas law

Therefore $y_i P = x_i P_i^{sat}$ **(2.1)**

$$\ln\left(P^{sat}\right) = A - \frac{B}{(T+C)}$$ Antoine equation **(2.2)**

The constants of the Antoine equation are given in Table 2.1.

Table 2.1.Antoine constants for the Ethanol-Water system(Coulson, Richardson & Sinnot, 1983) (Pressure in mm Hg and Temperature in Kelvin(K))

Compounds/ constants	**A**	**B**	**C**
Ethanol(1)	*18,9119*	*3803,98*	*-41,86*
Water(2)	*18,3036*	*3816,44*	*-46,13*

The composition of an ethanol-water mixture is then calculated as follows:

$$y_1 P + y_2 P = x_1 P_1^{sat} + x_2 P_2^{sat}$$

$$(y_1 + y_2)\, P = x_1\, P_1^{sat} + (1 - x_1)\, P_2^{sat}$$

(2.3)

$$P = x_1\left(P_1^{sat} - P_2^{sat}\right) + P_2^{sat}$$

$$x_1 = \frac{P - P_2^{sat}}{P_1^{sat} - P_2^{sat}}$$

$$x_1 = \frac{P - e^{A_2 - \frac{B_2}{T+C_2}}}{e^{A_1 - \frac{B_1}{T+C_1}} - e^{A_2 - \frac{B_2}{T+C_2}}}$$

$$x_1 = f(P, T)$$

Composition of the binary mixture of ethanol and water is a function of the system pressure P, and temperature of the system. Since in practical applications , the change in system pressure is small and within ± 1mm of Hg, it is assumed to be constant.

2.5 *Evaluation of ethanol-water composition using non-ideal temperature relations*

Using equations 2.1- 2.3, compositions of ideal mixtures can be calculated by the partial pressures of individual compounds. This is however not possible for ethanol(1) and water(2) mixtures, because of their non-ideal behaviour. This mixture forms a minimum boiling azeotrope at higher concentration ie at 90 mole% and 78,2 °C (Perry 1987), and therefore exhibits a positive deviation from Raoult's law. A relation between mole-fraction of ethanol in both the liquid and gas phase in the mixture is given in Figure 2.4. The azeotropic behaviour is evident from the figure where at 90 mole% ethanol the vapour and liquid compositions are equal.

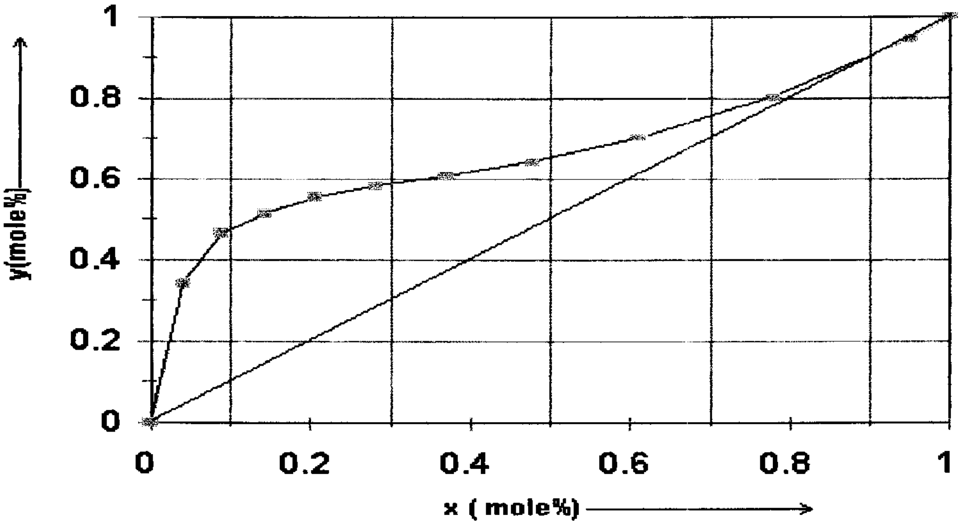


Figure 2.4 Mole fraction of ethanol in liquid phase (x) and gas phase (y) in ethanol- water mixture at atmospheric pressure of 760 mm Hg(International critical Tables,1930).

Due to its non-ideal behaviour, use of fugacity and activity coefficients influence the final calculation of composition of ethanol and water mixtures. A relation between fugacity (Smith & Van Ness, 1987) of a component i, and pressure of the component is given below in equation 2.4:

$$f_i = cP_i \qquad \textbf{(2.4)}$$

c for an ideal gas is a constant, equal to 1, and for a non-ideal gas c becomes the fugacity coefficient which is given below:

$$\phi_i = \frac{f_i}{P_i} \qquad \textbf{(2.5)}$$

Along with fugacity coefficients, activity coefficients also influence, composition determination in non-ideal mixtures. The activity coefficient is obtained from equation 2.6 –:

$$\gamma_i = \frac{\hat{f}_i}{x_i^1 f_i^0} \qquad \textbf{(2.6)}$$

Equation 2.1 is modified for the two components 1and 2 which incorporates ϕ and, γ as shown in equations 2.7.

$$y_1^1\ P = \frac{\gamma_1\ x_1^1\ P_1^{sat}}{\phi_1}$$

$$y_2^1\ P = \frac{\gamma_2\ x_2^1\ P_2^{sat}}{\phi_2} \qquad \textbf{(2.7)}$$

$$P = \frac{x_1^1 \gamma_1 P_1^{sat}}{\phi_1} + \frac{x_2^1 \gamma_2 P_2^{sat}}{\phi_2}$$

It is necessary to calculate the activity and fugacity coefficients for ethanol-water mixtures. This can then be applied when evaluating the composition of an ethanol-water mixture using the measurement of system temperature T.

a) *Calculations of fugacity coefficients for ethanol-water mixture*

In equations 2.7, ϕ_1 and ϕ_2 are functions of temperature, pressure and composition. In evaluating fugacity coefficients use is made of the parameters B^0 and B^1 given in equations 2.8 which are the second virial coefficients for a given gas and a function of reduced temperature only. Equation 2.9 gives the fugacity coefficient ϕ in which $T_r = T/T_c$ *and* $P_r = P/P_c$, and P_c, T_c are the critical pressure and temperature respectively.

$$B^0 = 0{,}083 - \frac{0{,}422}{T_r^{1{,}6}} \qquad \textbf{(2.8)}$$

$$B^1 = 0{,}139 - \frac{0{,}172}{T_r^{4{,}2}}$$

$$ln(\phi) = \frac{P_r}{T_r}(B^0 + \omega B^1) \qquad \textbf{(2.9)}$$

ω is defined as the acentric factor and is given by equation 2.10

$$\omega = -log_{10}(P_r^{sat})_{T_r=0{,}7} - 1{,}000 \qquad \textbf{(2.10)}$$

Table 2.2 Fugacity coefficients for ethanol water mixture (Smith & Van Ness, 1987)

	T_c (K)	**P_c(mm Hg)**	**ω**	**T (K)**	**P(mm Hg)**	**B^0**	**B^1**	**ϕ**
Ethanol	516,2	63,0	0,635	351,55	656	0,073	0,138	1,02 ≈1
Water	647,1	217,6	0,348	351,55	656	0,0108	0,137	1,01 ≈1

Table 2.2 gives values that are used in determining the fugacity coefficients for ethanol and water. Here ϕ_1 and ϕ_2 show 1-2% deviation from unity and their influence on calculations of composition using non-ideal equations 2.7 is negligible. Therefore they are assumed to be equal to1.

b) Calculation of activity coefficients for ethanol-water mixtures

γ_1 and γ_2 the activity coefficients, are independent of pressure but functions of temperature and liquid composition. These coefficients are positive for ethanol and water mixtures (due to positive deviation from the Raoult's law). Van-Laar equations 2.11 are used to determine γ_1 and γ_2 using the composition of ethanol and water respectively in the liquid phase, and parameters α and β. Values of α and β for ethanol and water mixtures are 2,203 and 0,7 respectively between 25-100 °C (Sandler, 1989).

$$\ln \gamma_1 = \frac{\alpha}{\left[1 + \frac{\alpha}{\beta}\frac{x_1}{x_2}\right]^2} \qquad \ln \gamma_2 = \frac{\beta}{\left[1 + \frac{\beta}{\alpha}\frac{x_2}{x_1}\right]^2} \qquad \textbf{(2.11)}$$

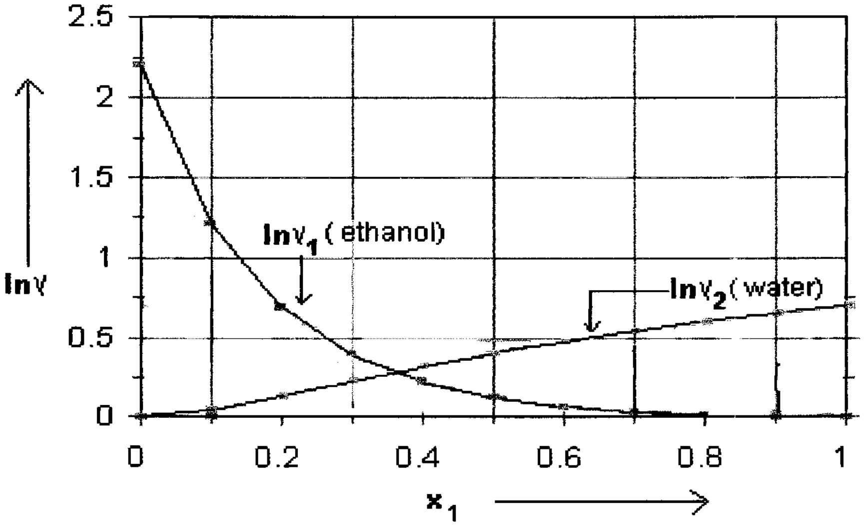


Figure 2.5 Relation between activity coefficients of ethanol and water mixture and mole fraction of ethanol in liquid phase (x)(Sandler, 1989).

The activity coefficients of both the components using equations 2.11 are presented as their natural logarithm values given in Figure 2.5.

(c) *Calculating the composition of ethanol using non-ideal equations.*

In determining the composition of ethanol using non-ideal equations the following steps were followed:

The composition of ethanol x_1 obtained from equation 2.3 for ideal mixtures was used to determine the activity coefficients γ_1, γ_2 from equations 2.12 a and b given below(derived from equation 2.11), where $x_2=1-x_1$.

$$\gamma_1 = e^{\frac{\alpha}{[1+\frac{\alpha x_1}{\beta x_2}]^2}} \qquad \textbf{(2.12 a)}$$

$$\gamma_2 = e^{\frac{\beta}{[1+\frac{\beta x_2}{\alpha x_1}]^2}} \qquad \textbf{(2.12 b)}$$

The activity coefficients calculated as a result are then substituted in equation 2.12 c(derived from equation 2.7).

$$x_1^1 = \frac{P - \frac{\gamma_2 P_2^{sat}}{\phi_2}}{\frac{\gamma_1 P_1^{sat}}{\phi_1} - \frac{\gamma_2 P_2^{sat}}{\phi_2}} \qquad \textbf{(2.12 c)}$$

As mentioned in section 2.5(a), ϕ_1 and ϕ_2 are assumed to be equal to 1. Therefore equation 2.12c results in equation 2.12 d.

$$x_1^1 = \frac{P - \gamma_2 P_2^{sat}}{\gamma_1 P_1^{sat} - \gamma_2 P_2^{sat}} \qquad \textbf{(2.12 d)}$$

This equation is used in calculating the composition of ethanol using non-ideal relations.

d) Comparing the composition obtained by ideal and non-ideal equations to a standard data

The calculated values of the composition of ethanol using both ideal equations (equations 2.3) and non-ideal equations (2.12 a-d) and the standard data from International Critical Tables (1930), at various temperatures (for reference) are shown graphically in Figure 2.6. The temperature range applied in the figure was 78-100 °C, which is the useful range for analysis.

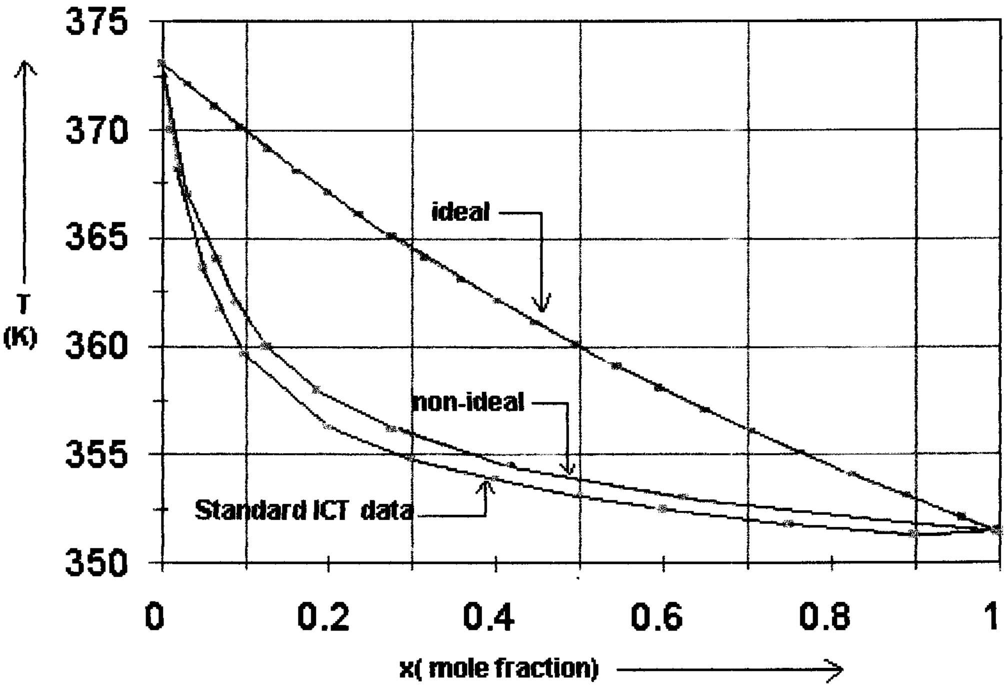


Figure 2.6 A relation between ethanol composition in liquid phase and compositions obtained from ideal, non-ideal T-C relations and standard ICT data for ethanol-water mixtures

2.6 The disadvantages of controlling the composition using temperature

It is disadvantageous to use temperature measurement for the purposes of inferring composition due to the following reasons:

(a)Inferential control is used when the controlled output of a processing unit cannot be measured directly.

In control practice, the success of this scheme depends heavily on the availability of a good estimator, which in turn depends upon how well the system, in other words description of non-ideal vapour and liquid in equilibrium, is known. Therefore, when the system is perfectly known, a perfect estimator can be constructed and consequently a perfect control is achieved. On the other hand, if the system is approximately known the inferential scheme provides control, of varying quality.

It is now necessary to determine to what extent the long derivation to infer composition (non-ideal) from temperature is inadequate. As it can observed from figure 2.6, a difference upto 2.3K exists for 20 % ethanol, which is large enough to show the difficulties in calculating compositions making use of non-ideal relations.

(b) It is also essential to show how inaccurate the calculated composition via temperature is especially in the region of the azeotrope. Ethanol and water system forms an azeotrope at 90% ethanol, as shown in Figure 2.4.

The temperature range is too narrow in this region ie 0,7 K, for the change in composition from 75% to 90% ethanol, also shown in Figure 2.6. Any slight error in temperature reading will cause a major error in the desired value of composition. Upon referring to the standard data (ICT) it is also observed that the relation between temperature and composition is non-linear as the composition increases from 75% to 100% ethanol, shown in Figure 2.7. Temperature drops from 78,7 ^{0}C to 78,2 ^{0}C for a change in composition from 75% to 90% ethanol whereas it increases from 78,2 ^{0}C to 78,4 ^{0}C when the composition increases from 90% to 100% ethanol.

It is difficult therefore to determine the composition of the mixture at higher concentrations of ethanol ie between 75-100% ethanol. The Figure 2.7 is an approximated quadratic response of a relation between higher concentrations of ethanol and their corresponding boiling point temperatures.

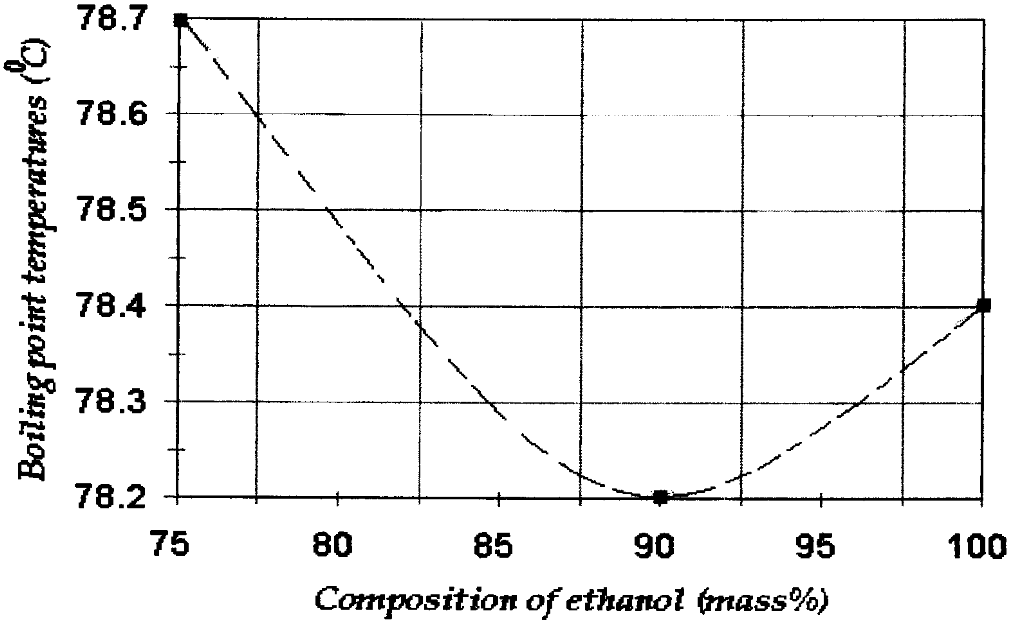


Figure 2.7 A relation between boiling point temperatures and mole% of ethanol and water mixtures for high concentrations of ethanol.

As such it is observed that inferential control using temperature as the sensing parameter has certain limitations in its use.

2.7 Capacitance Theories

Based on the limitations stated above, there is definite merit in investigating the feasibility of a capacitance cell to measure composition of ethanol-water mixtures, with a view to its adaptability for control of a distillation column. The main objective is to determine whether it will be advantageous to use the capacitance cell compared to the previously discussed measurement techniques namely chromatographs, infrared analyzers and temperature measurements. Before proceeding with details of such a device, the relevant theory of capacitors is covered in the material that follows.

2.7.1 *A relation between the dielectric constant of an ethanol-water mixture and its composition*

Figure 2.8 (Akerlof, 1932) shows a relation between the dielectric constant of ethanol-water mixtures in terms of its composition and temperature.

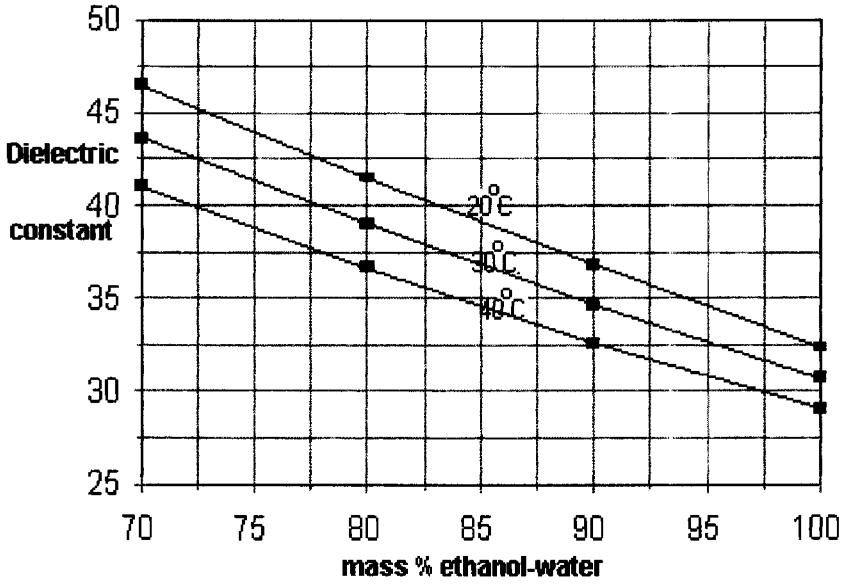


Figure 2.8 A relation between composition of ethanol and water mixtures and the dielectric constant

A maximum change of 15 units in dielectric constant is observed for each temperature in the range 70 -100 mass % ethanol-water, which is the typical operating condition of the composition measuring device to be constructed.

Dielectric constant is only influenced by two factors, the fractional composition and the temperature of the solutions as shown in the figure above.

2.8 Properties of Capacitors

This study looks at the theories involving the geometries of parallel plate and cylindrical capacitors. It is necessary to compare properties such as sensitivity and hold-up of the capacitors for evaluating a measurement system for purposes of control, since instrument dynamics would affect the performance of the control loop containing the instrument.

2.8.1 Parallel plate Capacitor:

A parallel plate capacitor (Cheng , 1989) consists of two parallel plates of cross sectional area S separated by a medium of uniform permittivity, ϵ, as shown in Figure 2.9.

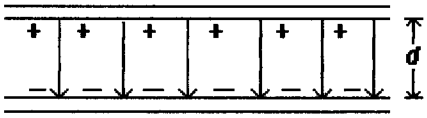


Figure 2.9 A Parallel Plate capacitor

The capacitance for a parallel plate capacitor if d is the distance between the plates is given as:

$$C_{pa} = \frac{\epsilon \; S}{d} \qquad \textbf{(2.13)}$$

Charges will be uniformly distributed over the conducting plates shown in the figure above.

2.8.2. Cylindrical Capacitor:

A cylindrical capacitor (Walker,1990) as shown in Figure 2.10 comprises of two concentric tubes having diameters a and b respectively. The space

i14767429
b14461067

between the conductors is filled with dielectric of permittivity ϵ. The length of the capacitor is L .

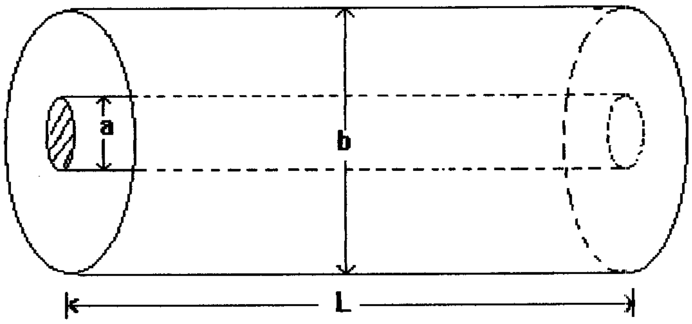


Figure 2.10 A Cylindrical capacitor

The capacitance of a cylindrical capacitor is given as :

$$C_{cy} = \frac{2\,\pi \epsilon L}{ln\,(\frac{b}{a})} \qquad \textbf{(2.14)}$$

2.8.3 Comparison of the performance of a parallel plate capacitor to a cylindrical capacitor

As mentioned earlier, system sensitivity and hold-up of each of the configurations should be compared to evaluate the performance of both capacitors. In the subsequent discussion, it will be shown that although cylindrical capacitors give more sensitive readings, smaller hold-ups in parallel plate capacitors enable them to be better suited for use in measurement systems.

Since dielectric constant is a function of liquid composition, one would prefer an instrument which is sensitive enough to provide a large change in capacitance for a given change in ϵ.

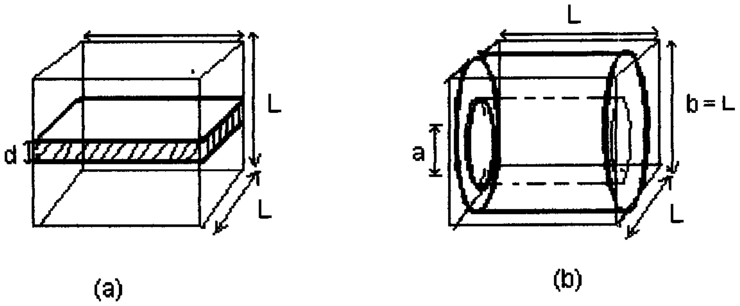


Figure 2.11 (a) A parallel plate capacitor placed in a cube of dimension L ; (b) A cylindrical capacitor placed in a cube of dimension L

An example of the two capacitors placed in a cube of dimension L is shown in Figure 2.11 given above.

The following assumptions were made to facilitate the calculations:

(a) b = L.

(b) b-a = 2d.

(c) (a) and (b) will also imply L -a = 2d.

(d) When the inner diameter a varies from 0 to L ; d will vary from L/2 to 0.

(e) The same liquid with dielectric constant ε fills the volume between the two capacitor configurations.

2.8.3.1 *Sensitivity Calculations*

The system sensitivity of the device is determined by the change in capacitance for a given change in the dielectric constant of fluid flowing through the device. Geometry of the capacitor determines this property. The sensitivities of both capacitor configurations are obtained to determine which of the two configurations results in a more sensitive device within the given geometrical constraints. Use of the example given in Section 2.8.3 and equations 2.13 and 2.14 are made in deriving equations 2.15.

$$C_{pa} = \frac{\epsilon S}{d} = \frac{\epsilon L^2}{d}$$

$$C_{cy} = \frac{2\,\pi\,\epsilon\,L}{ln\left(\frac{b}{a}\right)} \qquad \textbf{(2.15)}$$

$$C_{cy} = \frac{2\,\pi\,\epsilon\,L}{ln\left(\frac{L}{L-2\,d}\right)}$$

Since it is required that the sensitivities of both the capacitors be compared, $\partial C_{cy} / \partial \epsilon$, $\partial C_{pa} / \partial \epsilon$ were evaluated as equations 2.16. Reasons for using partial derivatives is because **d** in the relation is a variable.

$$\frac{\partial C_{pa}}{\partial \epsilon} = \frac{L^2}{d} \qquad \frac{\partial C_{cy}}{\partial \epsilon} = \frac{2\,\pi\,L}{ln\left(\frac{L}{L-2\,d}\right)} = \frac{-2\,\pi\,L}{ln\left(\frac{L-2\,d}{L}\right)} \qquad \textbf{(2.16)}$$

The ratio of $\partial C_{cy}/\partial \epsilon$ to $\partial C_{pa}/\partial \epsilon$ was compared at the two limits of d, 0 and L/2, to verify which of the capacitors gives a maximum change in capacitance for changes in the dielectric constants of the fluid. At the lower limit, this ratio is 0/0 ie indefinite. L-Hospital's rule is applied to this limit, which results in equations 2.17.

$$\frac{\frac{\partial C_{cy}}{\partial \epsilon}}{\frac{\partial C_{pa}}{\partial \epsilon}} = \frac{-\lim\limits_{d\to 0} \frac{d}{d(d)} \left(2 \pi d \right)}{\lim\limits_{d\to 0} \frac{d}{d(d)} \left(\ln\left(\frac{L-2d}{L} \right) L \right)}$$

$$\frac{\frac{\partial C_{cy}}{\partial \epsilon}}{\frac{\partial C_{pa}}{\partial \epsilon}} = \frac{\lim\limits_{d\to 0} -2\pi}{\lim\limits_{d\to 0} \frac{-2L}{L-2d}} \tag{2.17}$$

$$\frac{\frac{\partial C_{cy}}{\partial \epsilon}}{\frac{\partial C_{cy}}{\partial \epsilon}} = \pi \Rightarrow \frac{\partial C_{cy}}{\partial \epsilon} = \pi \frac{\partial C_{pa}}{\partial \epsilon}$$

The relations above show that, $\partial C_{cy}/\partial \epsilon$ is always greater than $\partial C_{pa}/\partial \epsilon$ at the lower limit. In the upper limit where d tends to L/2, the ratio of the sensitivities is 0 as given by equation 2.18:

$$\frac{\frac{\partial C_{cy}}{\partial \epsilon}}{\frac{\partial C_{pa}}{\partial \epsilon}} = \frac{0}{1} = 0 \Rightarrow \frac{\partial C_{cy}}{\partial \epsilon} = 0 \tag{2.18}$$

This value of the ratio at this limit is impractical because a sensitivity of 0 can never be useful in any measuring system. The ideal design of both types of capacitors applies to the condition when d →0, within practical limits. $\partial C_{cy} / \partial \epsilon$ is always larger than a $\partial C_{pa} / \partial \epsilon$ at this limit. The sensitivity of a cylindrical capacitor is therefore greater than that of a parallel plate capacitor.

2.8.3.2 Hold-up Calculations

It is interesting to observe how system hold-up influences measurement of a system. *Hold up* is defined as the time it takes to replace a given volume, given a constant flow rate in and out of the volume. It is the ratio of the volume of the vessel and the volumetric flow rate of the fluid in and out of the vessel. In control practice it is often necessary to minimise the hold up of a vessel.

Hold-up for each capacitor was calculated to determine which of the two capacitors gave a minimum time for measurement. If the volumes of cylindrical and parallel plate capacitors are given by V_{cy}, V_{pa} respectively, then:

$$V_{pa} = L^2 d$$

$$V_{cy} = \frac{\pi}{4} L\,(b^2 - a^2) = \frac{\pi}{4} L(b-a)(b+a) \tag{2.19}$$

$$V_{cy} = \frac{\pi}{2} d(\,b + a\,)L$$

A ratio of τ_{pa}, τ_{cy}, the hold up times of both the capacitors is given by equations 2.20.

$$\frac{\tau_{cy}}{\tau_{pa}} = \frac{V_{cy} \,/\, F}{V_{pa} \,/\, F} \tag{2.20}$$

$$\frac{\tau_{cy}}{\tau_{pa}} = \frac{\frac{\pi}{2} d(\,L + a\,)\,L}{L^2\,d} \Rightarrow \frac{\frac{\pi}{2}\,(\,L + a\,)}{L} \gg 1$$

It is observed that hold-up in a cylindrical capacitor is larger than a parallel plate capacitor, shown by the relation above. This implies that parallel plate capacitors give faster dynamics during measurements, explaining their choice.

2.9. Description of the circuitry (A.C and D.C capacitors)

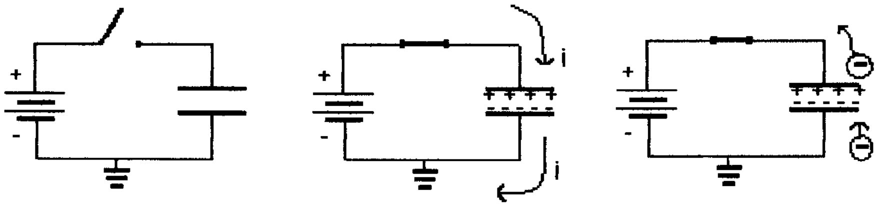
The capacitor used in this investigation , uses the variation of dielectric constant which is a function of composition as a means of sensing for measurement and control in a distillation column.

A capacitor has the ability to store electrical charge, and to increase its charge when the voltage across its terminals rises or to discharge when the voltage falls(Kip, 1965). In the following section, the behaviour of A.C and D.C capacitor circuits are discussed, to observe which of the two will be more suitable for measurement of composition.

2.9.1 Description of a D.C circuit connected to a capacitor

A D.C circuit(Kip , 1965) with individual capacitor plates is shown in Figure 2.12 (a) , (b)& (c). The supply current is constant before the switch is closed as shown in Figure 2.11 (a). With a D.C voltage source, current flows long enough to charge the capacitor . Then as the plates gain polarity and oppose additional charge , the charging current decreases until it reaches zero at the moment when the charge on the plates is equal to the voltage of the D.C source.

Distribution of positive and negative charges to the upper and lower capacitor plates respectively, will result as shown in Figures 2.12 (b) & (c).



(a) (b) (c)

Figure 2.12 An uncharged capacitor beginning to charge up after closing the switch. (Kip, 1965)

This capacitor behaves as an electrolytic cell with electrochemical reactions taking place at the two plates (ie the two electrodes) when a dielectric fluid like ethanol-water mixture is placed between the plates. The electrochemical reactions can be:

(a)Cathodic

The electrode in this case is stainless steel (Chapter 3, Section 3.1.2.2 (b)). Water in the mixture is subjected to a reduction reaction due to its ability to undergo the following reduction reactions (Stanley, 1985).

$$2\ H_2O + 2\ e^- \rightarrow H_2 + 2\ OH^- \quad - \qquad 0\ V \qquad \textbf{(2.21)}$$

(b) Anodic

Oxidation of the ethanol -water mixture due to removal of electrons to the electrode results in anodic reactions. This reaction gives hydrogen ions and carbon dioxide in the capacitor. The reaction is given by equation 2.22.

$$CH_3CH_2OH + 3\ H_2O - 8\ e^- \rightarrow 2\ CO_2 + 12\ H^+ \qquad + 1.09\ V \qquad \textbf{(2.22)}$$

The voltage of 5V in the circuitry of the measuring device (discussed in Chapter 3, Section 3.1.2) is larger than the individual half cell potential, sufficient to generate electrolytic reactions in the cell. Production of carbon-dioxide and hydrogen in the mixtures affect the dielectric constant in the solution, making it impossible to obtain a consistent, representative capacitance reading related to the composition of the liquid mixture.

D.C capacitors can therefore not be used in measurement systems, involving liquid composition determination of this kind.

2.9.2 *Description of an A.C circuit connected to a Capacitor*

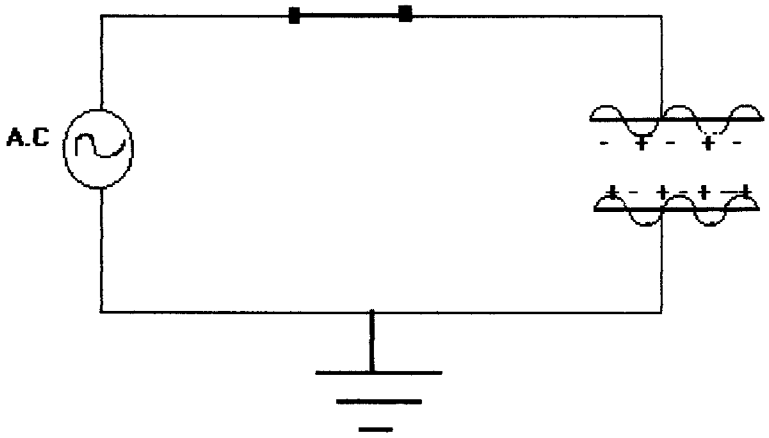


Figure 2 .13 An A.C input to a capacitor (Kip, 1965)

Current flow in an A.C circuit across a capacitor represents continuous and alternating charging and discharging of the capacitor plates, and no electron movement takes place directly between the plates. Therefore in an A.C circuit as shown in Figure 2.13, the voltage on a plate will vary sinusoidally from the positive to the negative charge. No nett charge forms on any of the plates, as a result. Electrochemical reactions similar to those in D.C circuits will therefore not be observed. It is therefore imperative to use an A.C circuit in any device that makes use of capacitance related measurements.

Chapter Three

Process Equipment

In this chapter a description of the process equipment used in the test setup is given as follows:

(a) The capacitance cell prototype and its associated electronics.

(b)The laboratory distillation column.

(c) Different A/D cards used in measuring voltage signals and D/A cards used in controlling the column.

3.1 Description of the capacitance cell and its associated electronics

3.1.1. The capacitance cell as a sensor(C7 , Figure 3.1)

As mentioned in the introduction, the purpose of the investigation was to develop and test a capacitance cell that provides direct measurement of composition of binary mixtures, in this case ethanol and water. A simple and reliable relation exists between the composition and dielectric constant of ethanol and water mixtures at a given temperature, as shown in Figure 2.7 . The previous chapter also provided the necessary background for using a parallel plate capacitor and an A.C circuit for capacitance measurements.

A prototype parallel plate capacitance cell was therefore fabricated to be used as a sensor based on the theory stated above. It is constructed of two rectangular stainless steel plates of dimensions 75mmX 50mm and which is used as the capacitor. A distance of 4mm was used between the plates. These dimensions were obtained from the work of Jannsen(1986).

3.1.2 Cell electronics

In order to obtain a reliable reading from the capacitor , substantial signal

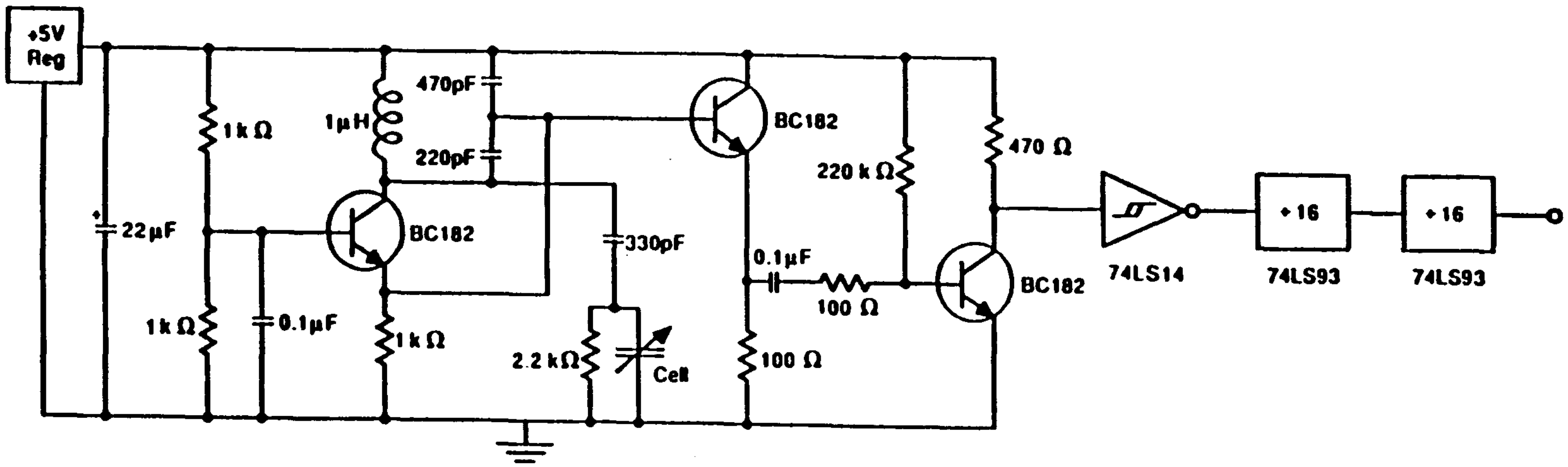


Figure 3.1 The original circuit diagram of the capacitance cell.

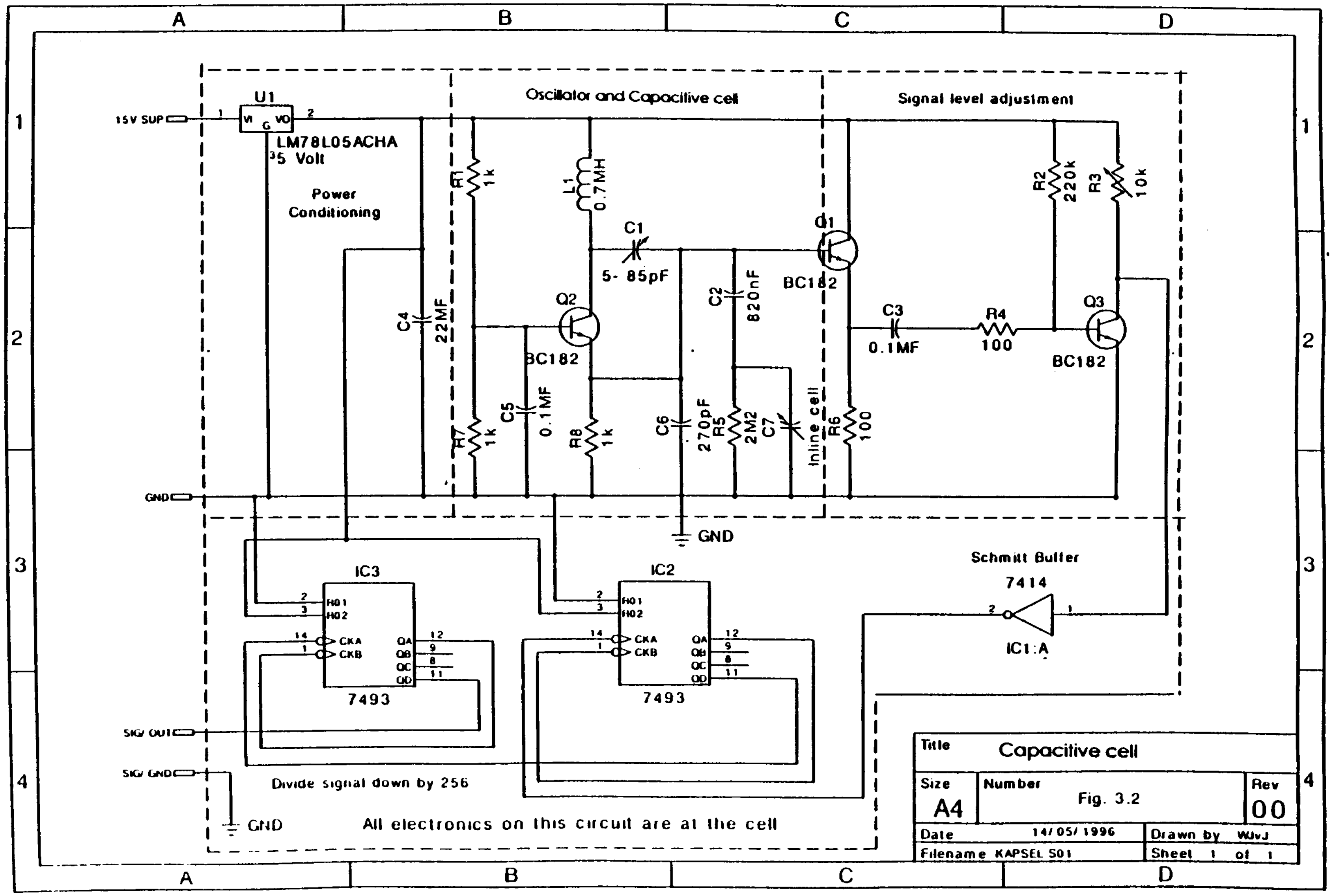


Figure 3.2 The modified circuit diagram of the capacitance cell.

conditioning was required. In the original circuit diagram Figure 3.1(Jannsen,1986), the oscillator and cell functions could not be divided. The capacitors 470 pF, 220pF(which were part of the oscillator), 330pF and the cell were connected across the supply line and the ground, providing a low impedance to the oscillator. This reduced the oscillator voltage to 0 V and stopped its functioning. Therefore the circuit Figure 3.1 was modified as shown in Figure 3.2 as follows:

a) The capacitors 470pF and 220pF were replaced by a tunable capacitor C6, and in order for the oscillator to provide the necessary oscillations, C1 was connected to C6.

b) The 330 pF capacitor was then replaced by 820pF capacitor for the purpose of stopping unwanted D.C current across the cell. Figure 3.2 shows the electronic circuit incorporating the capacitance cell.

The electronic circuitry in Figure 3.2 may be divided into four sections as given in block diagram Figure 3.3, the functions of each of these blocks is described here under:

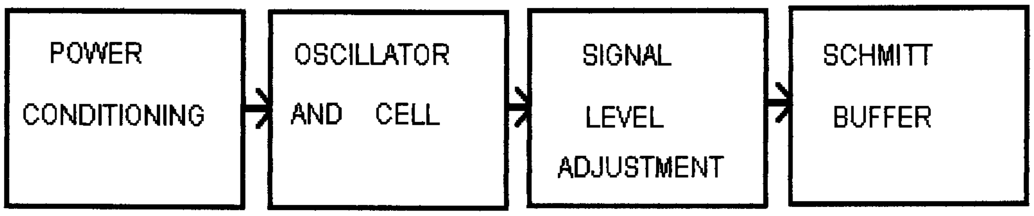


Figure 3.3 Block diagram showing different electronic components of the capacitance cell circuit

3.1.2.1 Power conditioning:

The *power conditioning unit* has the following functions:

(a) The operational voltage of the circuit in Figure 3.2 is 5V and the voltage from the D.C power source in the supply line is 15 V. The regulator U1 is used to reduce the voltage from the supply line to the circuit's operating voltage(Horowitz,1979).

(b)To provide a smooth and constant output current using a reservoir capacitor (which has a large capacitance) C4.

3.1.2.2 *Oscillator and cell :*

D.C current from the power conditioning section is supplied to this section. Since this section performs a very important role in generating an A.C current to the cell it can be described as the heart of the circuit. It has the following components :

a) L-C Colpitt's Oscillator.

b) The Cell, C7.

(a) L-C Colpitt's oscillator

It is the function of an oscillator (Rhea,1990) to produce a sinusoidal A.C current from the D.C supply current. This current is then supplied to the parallel plates to provide an effective capacitor for measurement purposes.

The configuration comprising of components Q2, L1, R8, C!, C6, C2 and R5 is the L-C Colpitt's oscillator. Since it is possible to tune a capacitor whereas an inductor cannot be tuned, its advantage in electronic circuit applications over the other type of oscillator, the Hartley's oscillator, is due to its two capacitors and one inductor configuration in comparison to the two inductors and one capacitor of the latter. This provides a better tuned circuit for measurement purposes.

(b) The cell prototype

This has already been described in section 3.1.1.

3.1.2.3 Signal level adjustment:

It is important to isolate the operation of the oscillator, because of components like the Schmitt trigger (IC1, in Figure 3.1) and frequency dividers IC2,IC3 which draw a certain amount of current from the circuit. This results in an unwanted drop in the voltage in the output signal. It is also necessary that the oscillator's output of 12 mV_{p-p} (voltage - peak to peak, referring to the difference in maximum and minimum voltage in an A.C sinusoidal signal) be amplified to 3 V_{p-p} because this enables switching on digital components such as the Schmitt Trigger and frequency dividers down the signal path. These components require higher voltages in the range of 1.5 - 4 V_{p-p} for their operation. The combination of the transistor Q1 and the resistor R6 is used for the dual purpose of acting as a buffer that protects the operation of the oscillator, and for magnifying of voltage output from the oscillator. Transistor Q1 will be a common collector with a high impedance input.

The A.C current in the output may not be influenced by any D.C current in the circuit. Resistor C3, the blocking resistor, blocks any D.C current resulting from the transistor Q1.

The Schmitt Trigger IC1, operates around a threshold voltage(described in the next section). Transistor Q3 and resistors R2, R4 and R3 will tune the output from the oscillator to adjust the level of the voltage to a threshold voltage.

3.1.2.4 Schmitt buffer

In the operation of the circuit a noisy output is produced which should be filtered out. A Schmitt trigger (Young, 1968) ,IC1, is employed for purposes of filtering this noise. After the resistor R3 has provided a threshold voltage, V as shown in Figure 3.4 the trigger will function as an on-off mechanism. Voltage values below this threshold are interpreted as 'off', and those above will be 'on'.

The 12 MHz output is a square wave and is analysed based on the width of its pulses. The width gives an indication of the frequency or period via the well-known relation **3.1** :

$$f = \frac{1}{T} \qquad \textbf{(3.1)}$$

A shorter period which is a higher frequency will indicate lower concentrations of ethanol while a longer period or lower frequency will show higher concentrations of ethanol.

The analogue-to-digital converter cards used in the laboratory, Eagle PC30 cards, cannot handle such high input frequencies and can read only voltage signals. Frequency dividers IC2, IC3 are therefore used to reduce the frequency output by a factor of 256 and frequency conversion to voltage is done using by a frequency-voltage converter(LM131 standard National Semiconductors device) to provide an input to this card. Turbo-Pascal was used as the interface programming language.

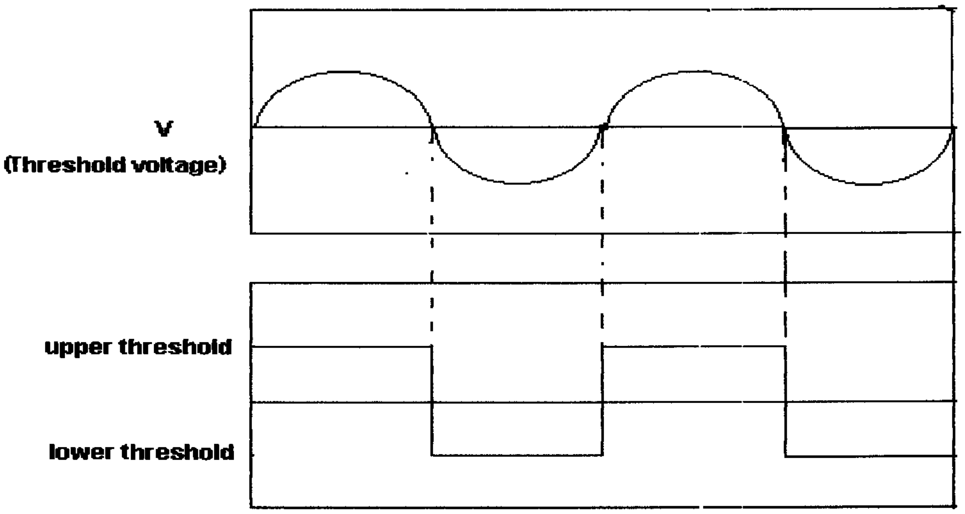


Figure 3.4 On-Off mechanism of a Schmitt trigger

3.2 Laboratory distillation column

The column given by Figure 3.5 and obtained from Van Niekerk (1995) has ten plates. Each plate is made of copper and has three bubble cap trays. The reboiler of the column of 20 litre capacity, consists of three, 2 kW electrical elements in parallel. Bottom product (B) tapped out of the reboiler and cooled by a heat exchanger flows into the collection drum, while the top product(D) flows into the reflux drum of 10 litre capacity. Liquid in the collection drum is pumped to the feed drum from where it is used as the feed to plate four of the column. The temperature in this drum is maintained constant using a single 2,3 kW heating element.

The liquid in the column is ethanol-water. It is cheap, non-toxic, non-corrosive and the boiling point can easily be reached in the laboratory. The azeotrope is reached at 90 mole % of ethanol at atmospheric pressure of 87 kPa. The following legend would describe the various components of the distillation column setup:

T1 : Feed drum
TC/1 : Temperature controller of the feed drum.
T1/I2 : Temperature indicator of the feed drum.
E3 : Heating element in the feed drum.
FI/1 : Feed flow rate (to the column)indicator
FC/1 : Feed flow rate (to the column)controller
KI : Feed stream valve
F : Feed
C1 : The column
CC/2 : composition controller cascaded to the thyristor(in the reboiler).

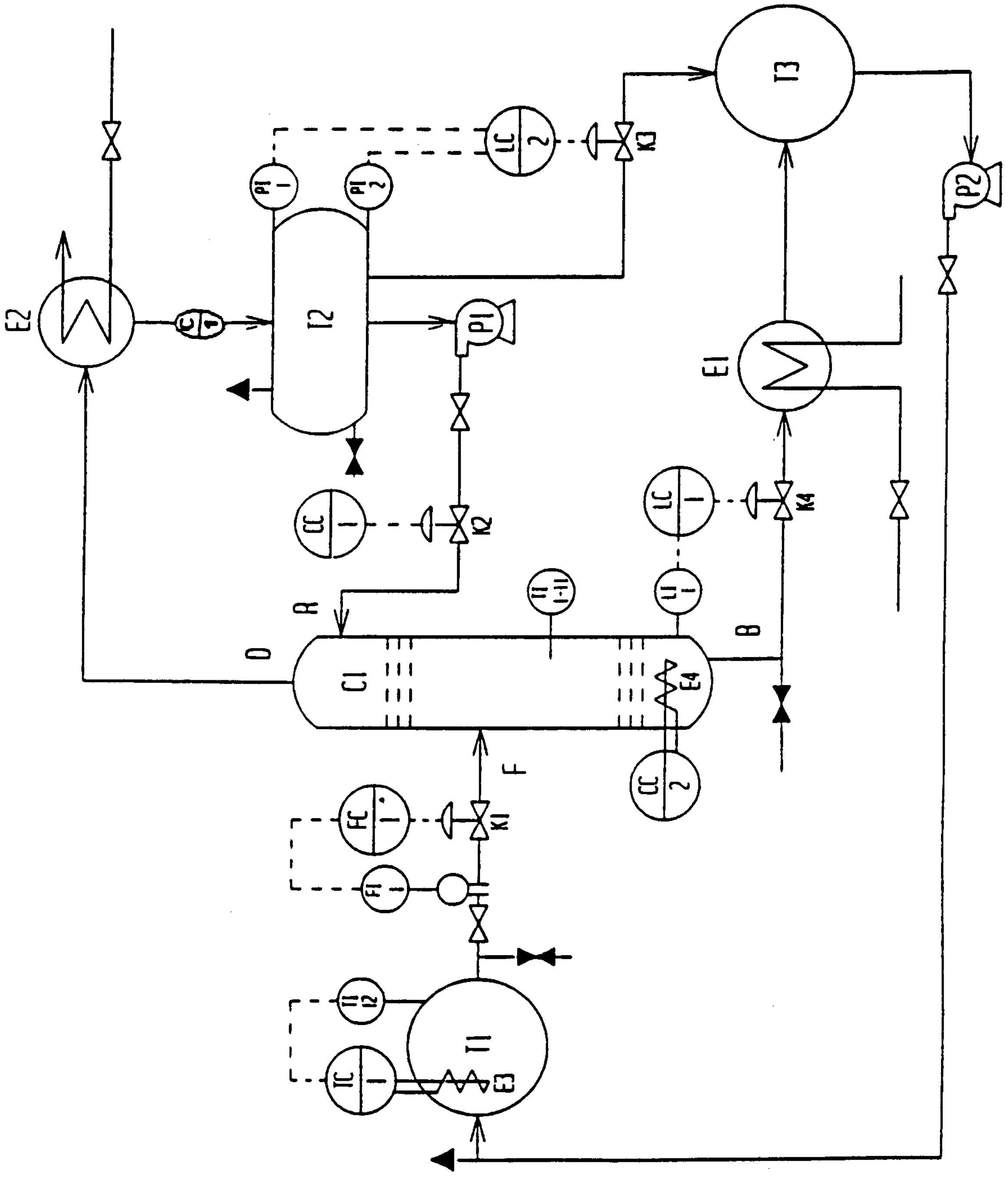


Figure 3.5 A schematic diagram of the distillation column setup.

E4 : Heating elements in the reboiler(The thyristor).
D : Distillate
R : Reflux
TI 1-11: Temperature indicators or thermocouples in the plates of the column.
CC/1 : composition controller cascaded to the reflux valve.
C/1 : composition analyser
K2 : The reflux valve
T2 : The reflux drum.
E2 : The condenser connecting top of the column to the reflux drum.
P1 : pump connecting the reflux drum to the top of the column via K2.
PI/1 & PI/2 : Liquid level indicators of the reflux drum.
LC/2 : level controller of the reflux drum.
K3 : distillate valve.
T3 : The collection drum.
P2 : pump connecting the collection drum to the feed drum.
LI/1 : liquid level indicator of the reboiler.
LC/1 : liquid level controller of the reboiler.
K4 : Valve connected to the level controller of the reboiler.
E1 : condenser connecting reboiler and the collection drum.
B : Bottom product

3.3 Data - converter cards

Reading of the analogue signals from the distillation column and the cell equipment was done by two types of cards namely the pc73 and the pc30 (Eagle Electric ,1992). The former reads temperatures from the column based on mV readings obtained from thermocouples, while the pc30 cards measure voltage signals.

Control signals to the column are provided using a pc66 card. The cards and the interface are calibrated to the correct limits. A brief discussion of the cards is given below.

3.3.1 *pc66 card*

The D/A conversions which are used for controlling the control valves and heat input to the reboiler by means of a thyristor were done by means of a pc66 card. This card has 12 output channels and a 12 bit accuracy.

3.3.2 *pc73 cards*

The two pc73 cards used in the equipment, obtain temperature readings in the distillation column using thermocouples which are K-type and have an accuracy of $\pm 0{,}5\ ^{0}$C. Eleven such thermocouples are placed, one each in the 10 plates and one in the reboiler of the distillation column shown in Figure 3.4 and provide mV- signals which are converted to temperature readings by the cards. The pc73 cards are equipped with a cold junction, enabling compensation of a reading due to variations in the ambient temperature. Each card has 8 channels with a 12 bit accuracy. The maximum rate at which the A/D conversion is done is 30 Hz.

3.3.3 *pc30 card*

The A/D conversions are done through 16 analogue single-ended input or 8 double-ended input, the maximum throughput being 30 kHz. Apart from this, the card has two 12 bit D/A outputs and two 8 bit D/A outputs and 24 digital I/O lines for D/A conversions, which are not used. The input range of this card is 0 - 10 V. It is used to measure the flow rates of feed, distillate, bottom product, reflux, thyristor heat input to the reboiler, and the capacitance cell output.

Chapter FOUR

Planning and execution of Experiments and Results

4.1 Experimental planning

Experiments were planned using the capacitance cell to achieve the following goals:

- To obtain a relation between the output from the capacitance cell and the composition of ethanol and water mixtures by calibration and to verify if the relation between the dielectric property and composition holds good for the chosen range of composition and temperature.

- To determine the dynamic lag of the capacitance cell. This was done by first studying the responses obtained from a first order process, in the context of its application on the column and the cell dynamic responses.

- To test the accuracy of the composition measurements on the column via capacitance cell readings and temperature measurements by comparison with the density method as reference for composition.

- To apply the capacitance cell on the column under both open-loop and closed loop conditions and study the effect of the capacitance cell on the dynamics of the column.

Experiments were conducted in the following manner:

An experimental setup was devised to obtain the relation between composition of an ethanol and water mixture and the output from the capacitance cell. It consisted of a circulating pump and a beaker containing the test mixture which was placed in a constant temperature bath and connected to the capacitance cell . Ten experiments at each of the following compositions, 75%, 85% and 90% were performed and the average of the corresponding voltage outputs were taken to obtain a final relation.

To determine the dynamic lag of the capacitance cell, five experiments were performed by changing the composition of the mixture flowing through the capacitance cell from 75 mass % to 85 mass% ethanol. This experiment was performed making use of the beaker setup of the previous experiment except that heating was not required. The dynamic lag obtained by these experiments would then be compared with the actual responses from the column to determine the effect of the capacitance cell dynamics on the column dynamics.

The capacitance cell was compact in size and therefore could be placed conveniently between the accumulator and condenser for providing in-line measurements to test the cell's behaviour on the column.

The steady state operating conditions of the column were:

(a) 50 % of ethanol-water in the feed, the feed temperature being 19-22 ^{0}C. However it was not possible to observe any difference in composition due to this small range of temperature variation. The fractional valve opening of the feed stream valve was 0,6 and feed flow rate was172,9 ml/sec.

(b) The fractional valve opening of the reflux valve was 0,5.

(c) Thyristor heat input to the reboiler: 50 % of its maximum power of 6 kW, ie 3kW.

(d) The steady -state composition of the ethanol-water mixture in the top product was 76.2 %.

The following experiments were performed on the column:

Accuracy testing involved changing the fractional opening of reflux valve by 0,25 , 0,35 and 0,45 from the steady state operating conditions. In this procedure, the density method was used as the reference to determine the accuracy of the measurements obtained by both temperature and capacitance methods.

A series of experiments were performed to determine whether the measurement of composition via capacitance measurement had any detrimental effect, ie, more dead time and lag, on the dynamic relation between top product composition from the column and an input change. At the same time, these measurements were compared to composition measurement via temperature to observe whether there was any substantial difference in the dynamics of these two techniques.

A series of experiments was also executed under closed loop conditions to confirm the capacitance cell performance.

4.2 Calibration of the capacitance cell

4.2.1 Calibration Process

Experiments were planned to calibrate the capacitance cell to determine a reliable relation between the output from the capacitance cell, and the composition of the ethanol and water mixture at a chosen operating temperature. The experimental setup is shown in Figure 4.1. In this setup, a constant temperature bath equipped with a heating coil and a beaker containing the ethanol-water mixture were used. A high speed pump circulated solutions at temperatures between 20-70 °C through the capacitance cell. PVC tubing was used to connect the various elements in this arrangement. Three different compositions 75, 85 and 90 in mass% ethanol and water mixture were prepared using volumetric flasks . The accuracy with which mixtures can be

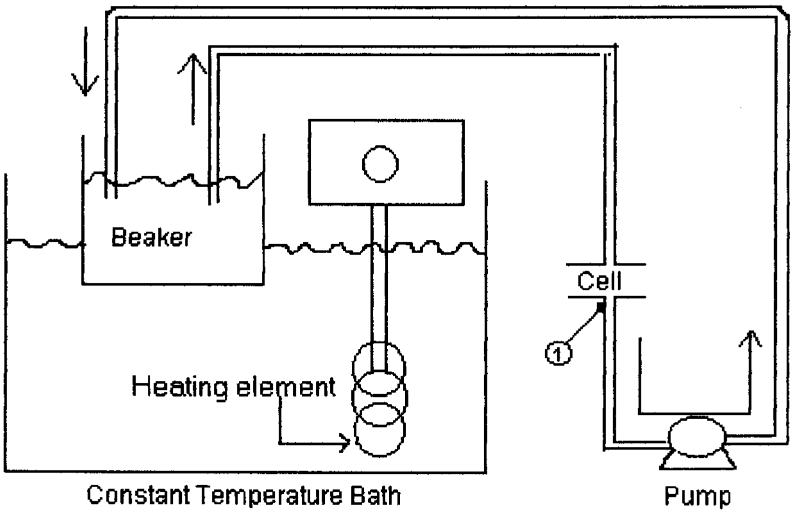


Figure 4.1 Experimental setup, calibration of the cell.

made up using volumetric flasks of 100ml solution is ±0,5%(Standard manufacturers specification 1107, South African Bureau of Standards). Taking into account that the specific gravity of 100% ethanol is 0,79 and that of water is 0,998 at 20 °C, the following mixtures were prepared.

75 mass% ethanol-water: 475 ml ethanol and 125 ml water.

85 mass% ethanol-water : 538 ml ethanol and 75 ml water.

90 mass% ethanol-water : 570 ml ethanol and 50 ml water.

The output was in millivolts (mV) obtained as result of frequency-to-voltage conversion, the frequency signal from the capacitance cell which in turn was a function of ethanol composition (also explained in section 3.1.2.4). The mV output was measured using a pc30 card, and the chosen operating temperature maintained by monitoring temperature reading through a thermocouple placed close to the cell (position 1 in Figure 4.1) in the flow stream , the reading obtained from a pc73 card.

During initial experiments, the readings from the capacitance cell were not repeatable. A trouble-shooting exercise revealed the following :

(a)Bubbles started forming at temperatures over 40°C, due to the onset of boiling and generation of vapour bubbles (Smith & Van Ness, 1987). The bubble formation caused a variation of the dielectric constant of the solution. The output from the capacitance cell was never consistent because of this. This problem was solved by ensuring that the feed temperature of the liquid to the cell is in the range of 20-40°C.

(b)Formation of a white precipitate was observed during the initial test runs and this was attributed to the presence of plasticisers on the PVC that were extracted by ethanol at high temperatures. PVC tubing was therefore replaced by silicon tubing.

(c)The high speed pump was replaced by a low speed circulating pump after it was observed that the cell needed smooth running conditions in order to provide accurate results.

(d)A drift in the output readings from the cell was observed due to the components of the cell electronics, which were temperature sensitive. An air conditioner used in the laboratory provided a constant external temperature in the range of 20-21°C and solved this problem.

(e)The cell electronics required a certain amount of time to reach steady state after switching on, to be able to give consistent readings. This time was found to be approximately 4-5 hours.

4.2.2 *Calibration results*

After overcoming the difficulties stated above the output was consistent. Ten experiments were performed at three different temperatures 20 °C , 30 °C , 40 °C, for each composition, 75 %, 85 %, 90 %. An average of the mV output of the 10 experiments, resulted in Figure 4.2.

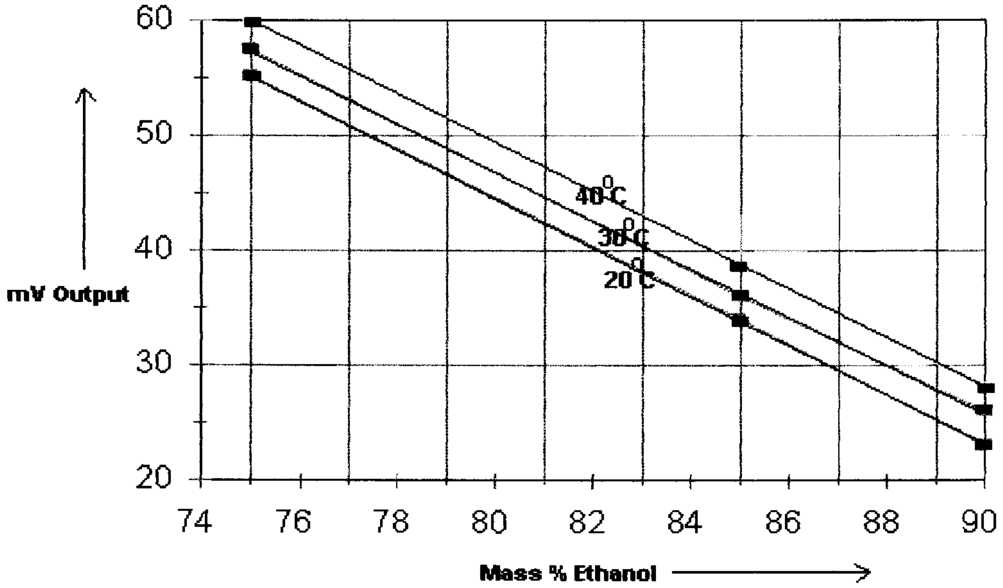


Figure 4.2 A relation between composition of ethanol and water mixture and mV output

A spreadsheet was used to obtain a linear regression of mV readings as a function of temperature and composition. The least squares method was applied and the relation is given as equation 4.1. The correlation coefficient for this curve fit was 0,999.

$$\mathbf{mV := -2{,}128\ C + 209{,}83 + 0{,}24(T)} \qquad \mathbf{(4.1)}$$

Rewriting equation 4.1 to obtain an expression for composition as function of mV output and temperature as shown below.

$$\mathbf{C := -0{,}47mV + 0{,}113\ (T) + 98{,}62} \qquad \mathbf{(4.2)}$$

This expression will be useful to calculate the unknown composition of a sample of ethanol and water mixture, when the readings of the mV and temperature are known.

Using equation 4.2, the derived values of composition obtained from the 10 experiments is calculated and presented in appendix I for temperatures of 20 °C,30 °C °C,40 °C respectively.

Tables 4.1-4.3 show the mean value and the confidence limits or the standard deviation of the derived composition from these experiments. A confidence limit is defined as the range in which the mean of an experimental data is accurate, and the standard deviation gives the confidence limits of a certain set of data(Manahan, 1989). From Tables 4.1-4.3 it is observed that the confidence limits are within ±0,55 which is good enough for all practical purposes.

Table 4.1 Statistical data of the calibration procedure at 20 °C

Composition	Mean	Standard Deviation
75%	75,03	± 0,542
85%	84,9	±0,383
90%	90,07	±0,53

Table 4.2 Statistical data of the calibration procedure at 30 °C

Composition	Mean	Standard Deviation
75%	75,22	±0,542
85%	85,09	±0,542
90%	89,79	±0,55

Table 4.3 Statistical data of the calibration procedure at 40 ^{0}C

Composition	Mean	Standard Deviation
75%	74.94	± 0,52
85%	85,75	±0,55
90%	90,02	±0,53

4.3 Determining dynamic process parameters for a first order process after providing a step input.

Response of a first order process after providing a step change in the input as shown in Figure 4.3 (a), resembles Figure 4.3 (b).

In the response curve Figure 4.3 (b), y is the output, τ_p represents the time constant of the process for the column for the change, τ_d the process dead time, K_p the process gain which gives the ratio of the change in the magnitude of the output(K_r) to the change in the magnitude of the input(K_s)

Therefore $K_p = K_r / K_s$. Since the response is assumed to be that of a first order process, at 0,632 K_s on the y-axis, the corresponding reading on the x-axis gives the time constant for the process, τ_p.

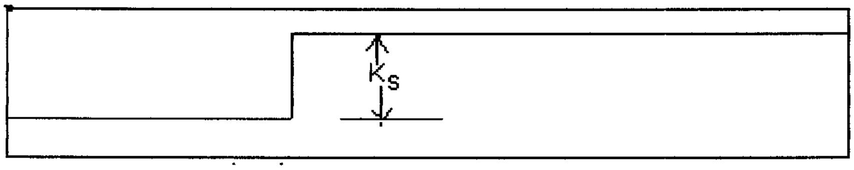


Figure 4.3 (a) Step input to a system

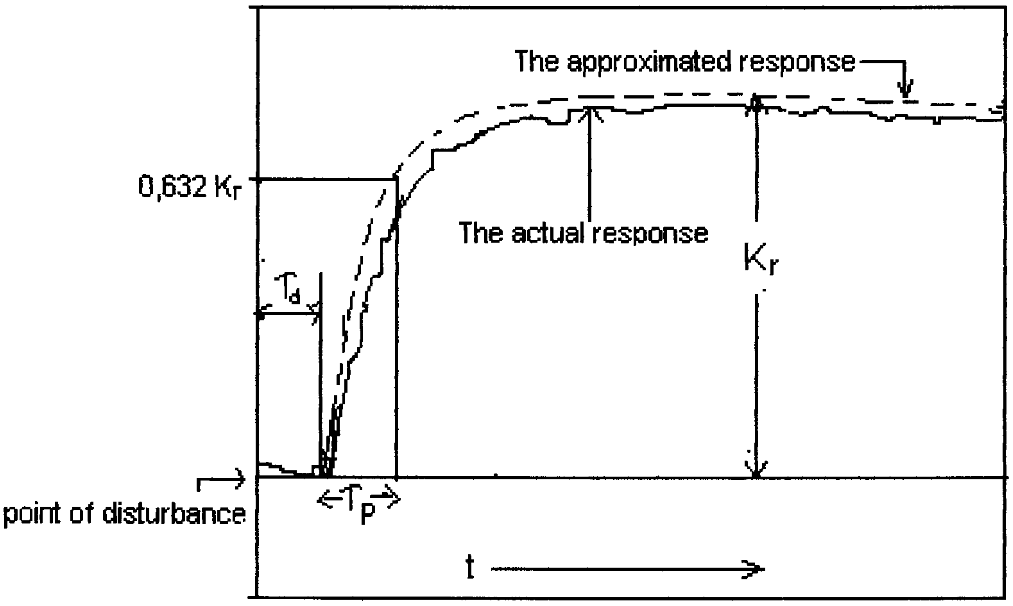


Figure 4.3(b) First order response of a system for a step change in the input

4.4 Cell dynamics lag testing

Dynamic lag for the capacitance cell gives an indication of how quickly the capacitance cell reacts for a change in composition.

This experiment was performed by taking two beakers one containing 75% ethanol and the other containing 85% ethanol. Then using the arrangement in Figure 4.1 and removing the heating equipment(heating is not required for this experiment), solution from the beaker containing 75% ethanol is pumped through the capacitance cell. After this the tube was placed in the beaker containing the 85% ethanol solution and the response reached its new steady state value within ±5%. The response time of the cell was determined using a sampling interval of 0,1 second.

Five such experiments were performed and the time for the change in the composition is given in Table 4.4 below. The largest response time for the change in composition was 1,3 seconds and the response of the capacitance cell was observed at this value, as shown in Figure 4.4. This response was assumed to be first order because the capacitance cell acted as the only single medium between the pumped solution and the reading obtained (Stephanopoulos, 1984). The corresponding dynamic lag was therefore obtained similar to Figure 4.3(b) and was 0,4 seconds, also shown in Figure 4.4.

Table 4.4. Dynamic lag of the capacitance cell

	Expt 1	Expt 2	Expt 3	Expt 4	Expt 5
Response Time (sec)	1 sec	1,2 sec	1,3 sec	1,2 sec	1,1 sec
Dynamic (τ_p) Lag	0,2 sec	0,3 sec	0,4 sec	0,3sec	0,2 sec

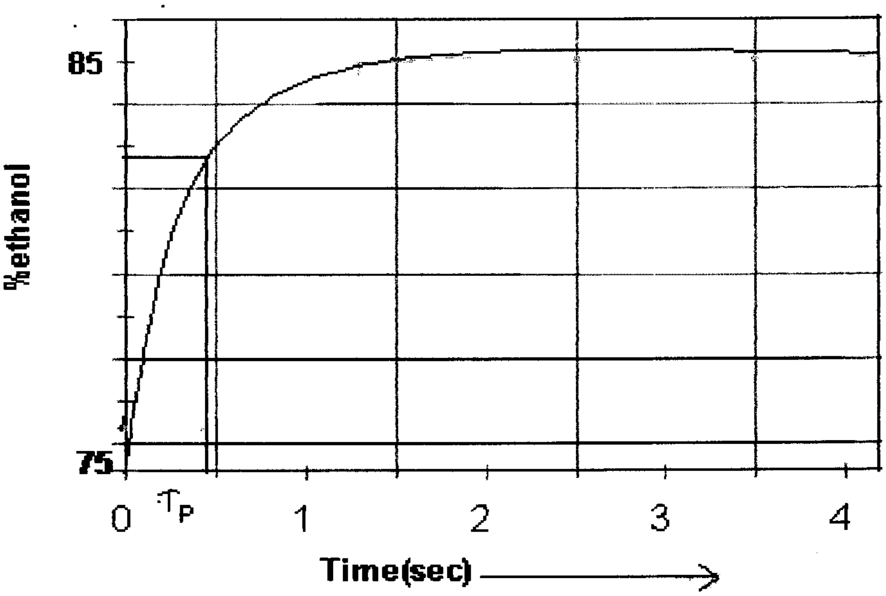


Figure 4.4 Dynamic lag of the capacitance cell for response time of 1,3 seconds

4.5 Determination of the accuracy of the capacitance cell.

4.5.1. Methods to determine accuracy.

It was planned to test the capacitance cell for accuracy by using a reliable reference and comparing the readings obtained by the capacitance measurement technique.

Accuracy may be expressed by the following expression

% Accuracy of a measurement technique=100- { A-B}/{ B} * 100 (4.3)

where A= composition determined via the new measurement technique.

B= composition determined via the reference testing method .

Density determination was used as the reference method for confirming the accuracy of readings of the capacitance cell. The relation between density of the mixture and composition is shown in Figure 2.3(Perry,1987). The accuracy of determining density of a sample is ± 0,00001 g/mL using the gravimetric analysis (Manahan.S.E, 1986).

In the following paragraphs, results of accuracy testing are presented.

4.5.2 Accuracy testing for temperature and capacitance methods.

In order to compare readings via the two available techniques, ie via temperature measurement and via capacitance measurement, the values so obtained were compared with composition obtained by means of density measurement. From these readings it was desired to obtain the accuracy of the capacitance cell readings as installed, compared with that of temperature measurements. The temperature measurements were performed by placing a thermocouple on the top tray due to reasons mentioned in section 2.4. Equations 2.1 -2.13 were used to obtain composition measurements by means of temperature, while equation 4.2 was used to obtain composition readings by means of capacitance measurements. These equations were introduced in an algorithm for determining composition of ethanol and water mixture. Density measurements involved the weighing of a sample of the top product in constant volume flasks, then obtaining the corresponding composition reading based on the density value and using of Figure 2.3.

The pc30 and pc73 cards were used to obtain temperature and capacitance measurements using a sampling interval of one second. Composition was obtained by density measurements using 10 ml constant volume flasks, the accuracy being ± 1%. The top product sample was taken at 20 ^{0}C and weighed using a mass balance, the accuracy of these measurements being ± 0,001% (Standard manufacturers specification 1107, South African Bureau of Standards). Density was calculated at 20 ^{0}C and composition of the sample was obtained from Figure 2.3.

To determine the accuracy of the capacitance and temperature methods step disturbances to the reflux valve(refer to Figure 3.2) of magnitudes 0,25, 0,35 and 0,45 was applied. The density and capacitance measurements were performed after the vapour of the top product is cooled down by the condenser, therefore lag between these measurements was nil. Since the temperature measurements were performed on the top plate there is a time lag in measurement between temperature and capacitance methods. The lag was between these two methods was found to be 7 seconds for 0,25 change, 6.5 sec for 0,35 and 6 seconds for 0,45 changes in the reflux valve positions. These lags were compensated for in calculating the accuracy of the measurement techniques.

After obtaining the raw data, the average of readings of both temperature and capacitance measurements in a 50 second interval were determined while at the same time taking a sample of the distillate in this interval. By performing this, it was intended to use the average deviation from the actual value for the capacitance and temperature methods for purposes of determining accuracy of these techniques.

Figures 4.5, 4.6 and 4.7 show the results obtained by the three reflux valve changes. Each composition point is represented by the average value obtained in a 50 second interval. It can be observed from the figures that the measurements from the capacitance cell are closer to the density method. NB. In this investigation, the responses from the column are given as a deviation from the steady state composition (ie 76,2% ethanol).

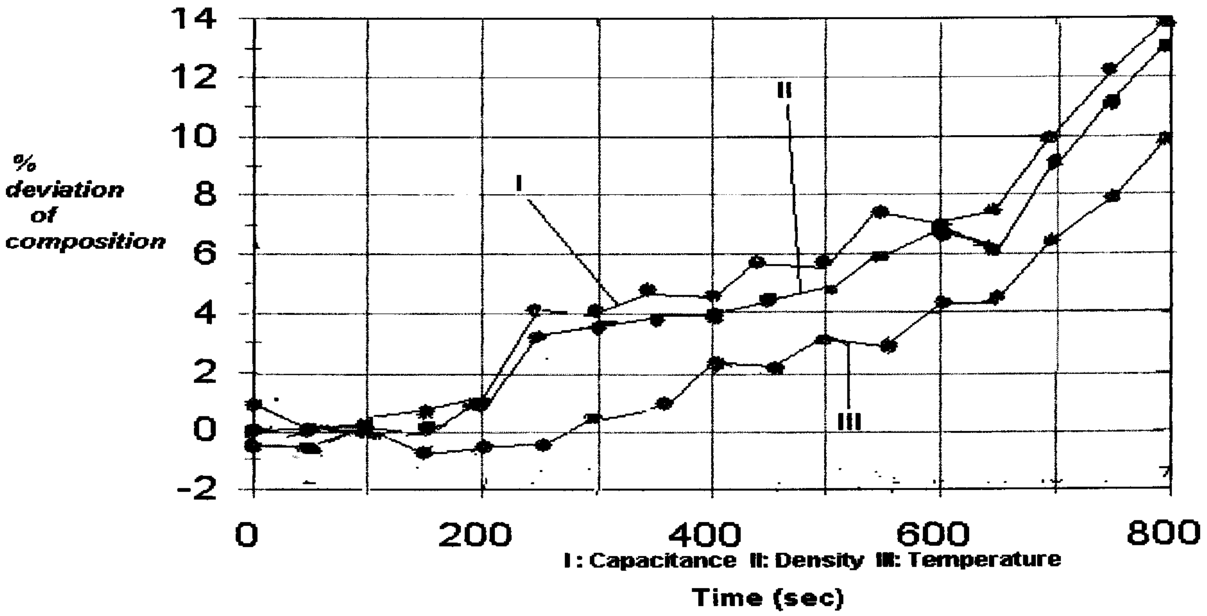


Figure 4.5 Comparison of composition via capacitance, temperature and density measurements for a change in the reflux valve by 0,25 units

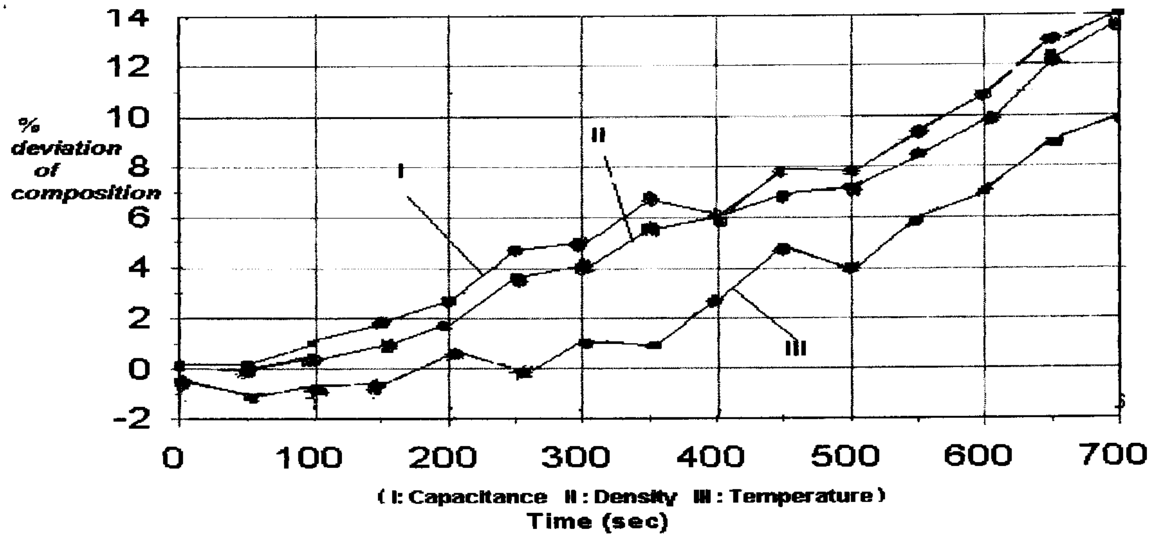


Figure 4.6 Comparison of composition via capacitance, temperature and density measurements for a change in the reflux valve by 0,35 units

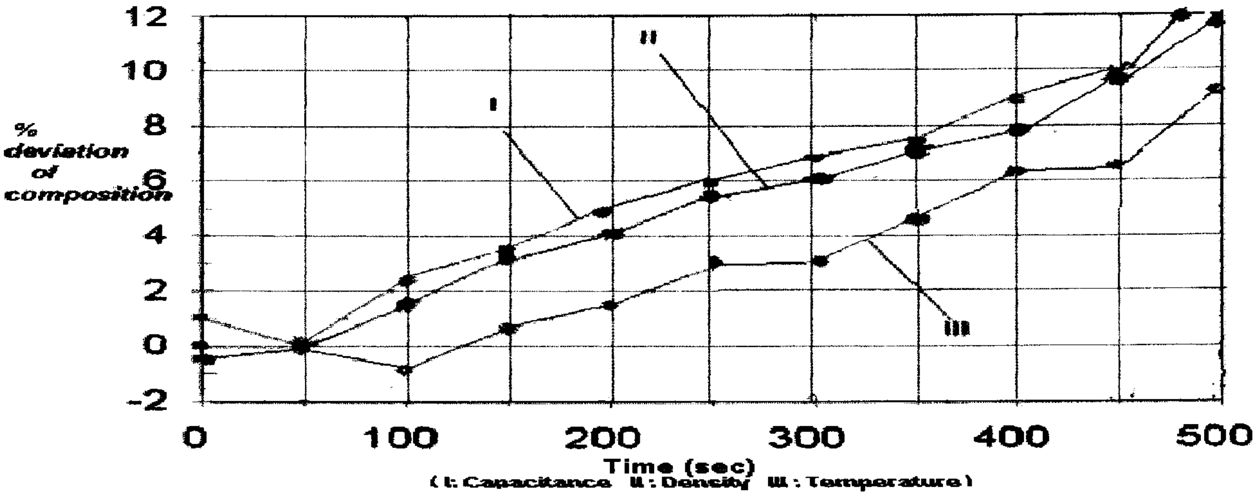


Figure 4.7 Comparison of composition via capacitance, temperature and density measurements for a change in the reflux valve by 0,45 units

4.6 Determining dynamic process parameters for the column

In order to control the top product composition of the distillation column, dynamic models relating process outputs to various inputs were obtained from the column. This was done by means of step tests. The dynamic model parameters ie steady state gains, process time constants and dead times were determined from the response curves. Two important disturbances were used namely, change in the power supplied to the reboiler and change in the position of the reflux valve. The responses obtained as a result of the changes were assumed to be first order. This is because in applying the change from the thyristor for example, each plate of the column can be assumed to add a lag, resulting in a higher order system (definitely greater than a second order system). This higher order system can in turn be considered as a first order system with dead time.

4.6.1 Model responses with change in the position of the reflux valve in both the measurement systems.

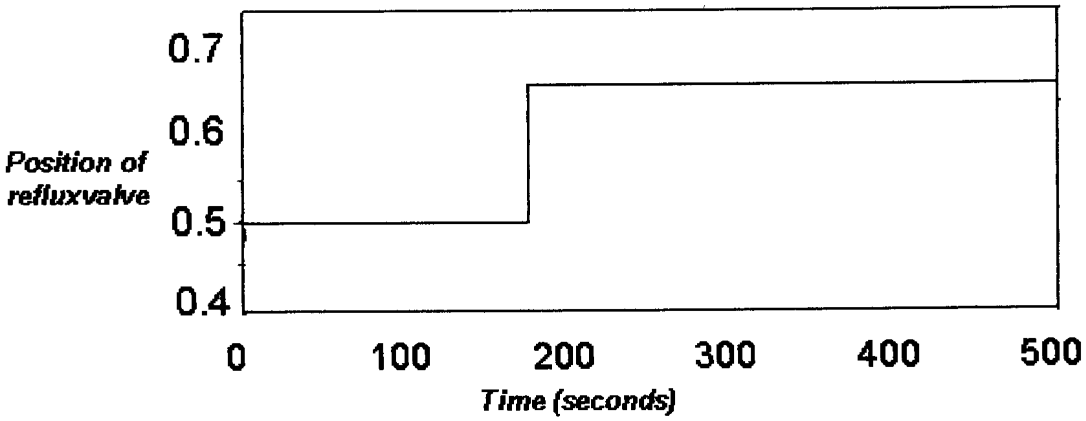


Figure 4.8 (a) A change in the reflux valve position by 0,15 units.

A reflux valve change of 15 % was applied, as shown in Figure 4.8 (a) to obtain a dynamic model for the effect of this on top product composition. The responses were assumed to be first order by both temperature and capacitance measurements, as shown in Figures 4.8(b) and 4.8(c). The first order discrete models using regression analysis(Stephanoupoulos, 1984)for both the methods were :

Temperature method(Figure 4.8 (b)): $y_n = 0{,}96 * y_{n-1} + 0{,}12 * m_{n-1}$

Capacitance method(Figure 4.8 (c)): $y_n = 0{,}96 * y_{n-1} + 0{,}13 * m_{n-1}$

These responses were filtered in Matlab (procedure is described in appendix III) using single exponential smoothing (Seborg , Edgar &Mellichamp,1989). In this method the equation 4.4 was used:

$$y_n = \alpha * x_n + (1-\alpha) * y_{n-1} \qquad (4.4)$$

The values of α for the filtered values of both temperature and capacitance methods were 0,105 and 0,109 respectively, the correlation coefficients between the filtered and unfiltered readings were 0,982, and 0,981 respectively.

Since the responses were assumed to be first order, Figures 4.3(a) and (b) are used to obtain the dynamic model parameters. The responses indicated a change in the composition of 4,2 and 4,7 respectively, besides different time constants and dead times, also shown in Table 4.5.

Table 4.5 Model parameters of the column after applying a change in the reflux valve position

Temperature measurements	K_p = 28 mass % / fractional valve opening	τ_p = 55 sec	τ_d =17sec
Capacitance Measurements	K_p = 31,33 mass % / fractional valve opening	τ_p =70sec	τ_d = 25sec

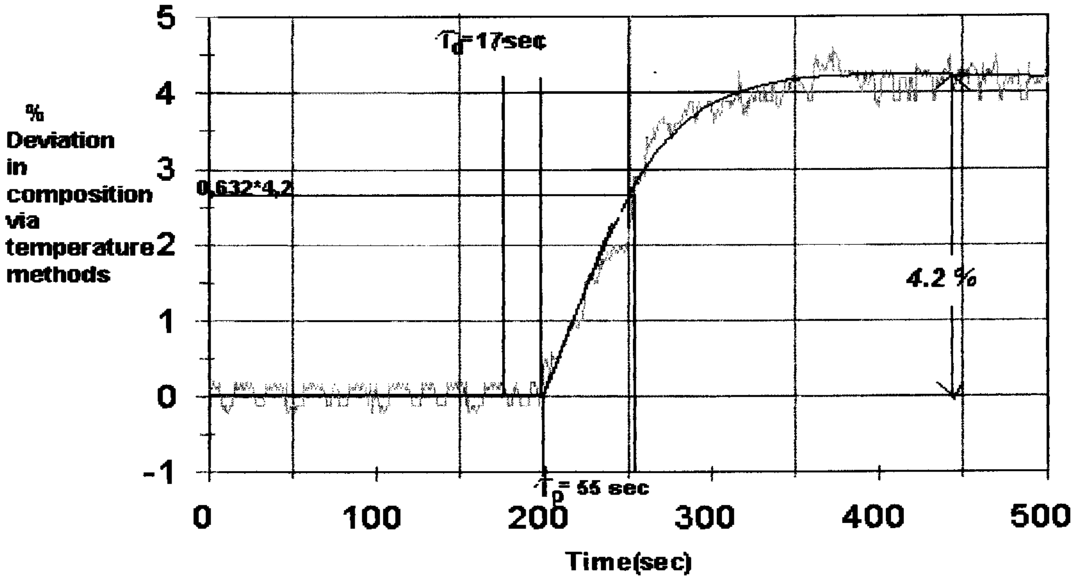


Figure 4.8 (b) Model response of top product composition with a change in 0,15 units in the position of the reflux valve by temperature measurements.

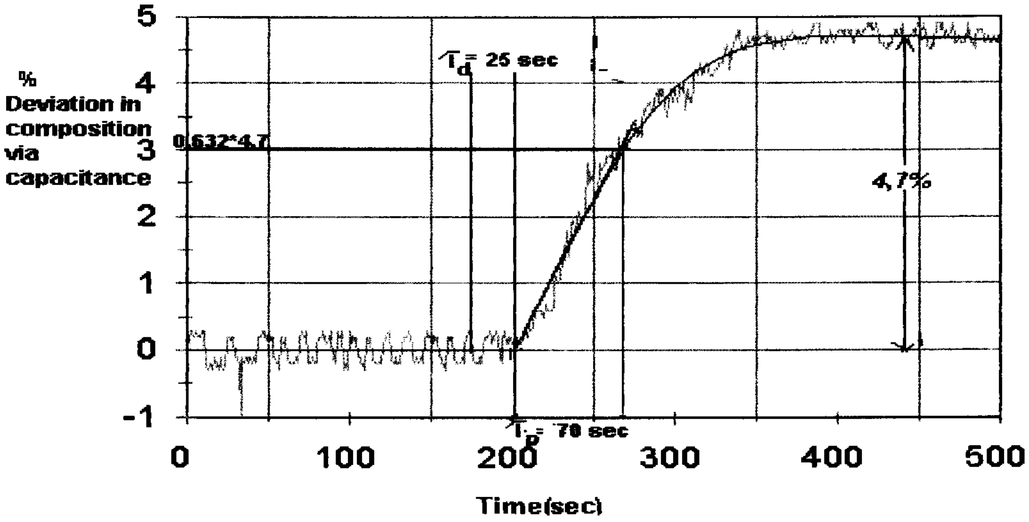


Figure 4.8 (c) Model response of top product composition with a change in 0,15 units in the position of the reflux valve by capacitance measurements.

The gain of the process was obtained as follows using the method described in section 4.3:

Kp= 4,2/0,15= 28 %mass/ fractional valve opening (Temperature measurements)

Kp= 4,7/0,15= 31,33 %mass/Fractional valve opening (Capacitance measurements)

4.6.2 Model Responses with change in the heat input to the reboiler.

Step changes to the reboiler by applying a 50% increase in the heat input, as shown in Figure 4.9 (a), resulted in first order responses given in Figure 4.9(b) and Figure 4.9(c) by the respective measuring methods.

The composition changes in the top product by temperature and capacitance methods were -1,25 and -1,37 and the corresponding process time constants and dead times are shown in Figures 4.9 (b) and 4.9(c) and Table 4.7.

The first order discrete models given by regression analysis(Stephanoupoulos, 1984)for both methods are as follows:

Temperature method: $y_n = 0{,}98 * y_{n-1} + 0{,}001 * m_{n-1}$

Capacitance method: $y_n = 0{,}97 * y_{n-1} + 0{,}001 * m_{n-1}$

These responses were filtered in Matlab (procedure is described in appendix III) using single exponential smoothing(Seborg, Edgar &Mellichamp, 1989). This method makes use of equation 4.4. The values of α used to obtain filtered values of temperature and capacitance methods were 0,2 and 0,23 respectively, and the correlation coefficients between the filtered and unfiltered readings were 0,983 and 0,986 respectively.

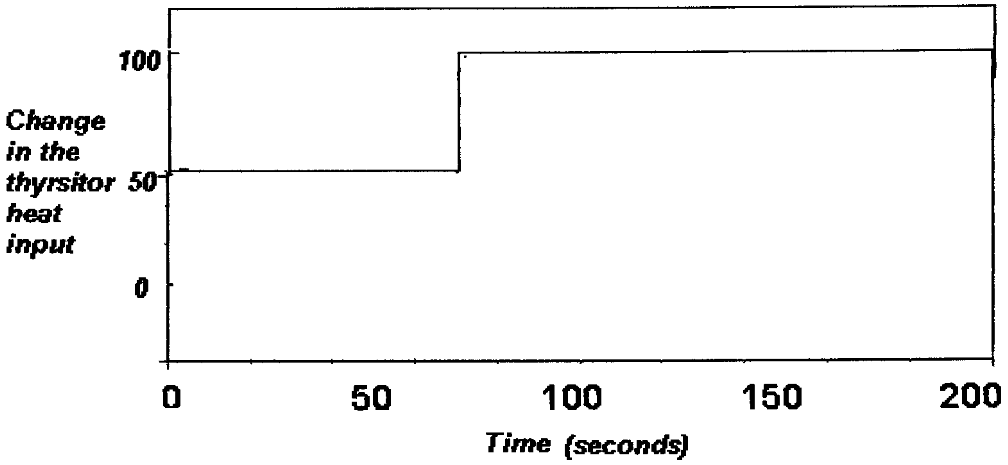


Figure 4.9(a) A change in the thyristor by 50 % from the operating conditions

K_p = -1,25/50 *100 = -2,5 %mass/%thyristor input(Temperature measurement)

K_p = -1,37/50 *100 = -2,75 % mass / % thyristor input(Capacitance measurement)

Table 4.6 Model parameters after applying a change in the thyristor

Temperature measurements	K_p = -2,5 mass %/ % thyristor heat input	τ_p =30 sec	τ_d = 12 sec
Capacitance Measurements	K_p = -2,9 mass %/ % thyristor heat input	τ_p = 35 sec	τ_d = 14 sec

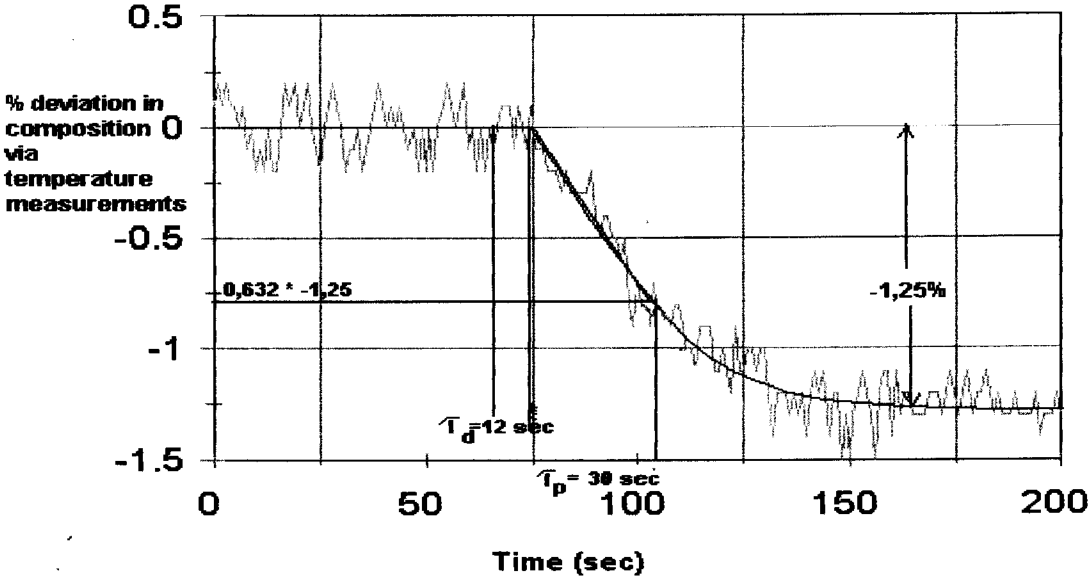


Figure 4.9 (b) Model response of top product composition with 50 % change in the thyristor by temperature measurements.

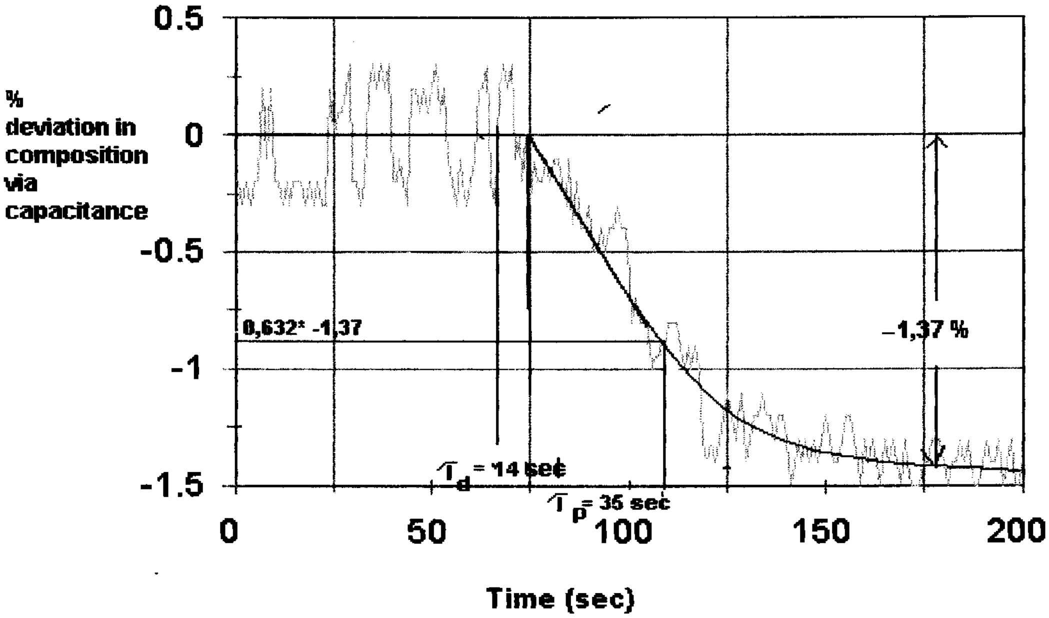


Figure 4.9 (c)Model response of top product composition with 50 % change in the thyristor by capacitance measurements.

4.7 Closed-loop control of the column using the capacitance cell.

Experiments with closed loop control of top product composition were conducted on the column using the capacitance cell, the purpose of these experiments being to confirm whether the capacitance cell works satisfactorily under closed loop conditions. In order to confirm this, controllers were tuned and a 1% change in the setpoint ie changing the composition from 76,2% to 77,2%, was applied. The reflux valve was used as the manipulated variable in one series of experiments and the the boil-up rate(thyrsitor) as the manipulated variable in another series of experiments and the responses were monitored. The parameters of the controller by using the reflux valve and the thyristor in separate sets of experiments are given below in Table 4.8:

Table 4.8 Controller parameters using both Reflux valve position and thyristor as final control elements.

Reflux valve position	K_c = 0,05755 fractional valve opening/ % mass	τ_I = 109,821sec
Thyristor	K_c = -0,8055 % thyristor input/ % mass	τ_I =47,346sec

A PI controller action was applied and the responses for both the manipulated variables, are shown in Figures 4.10 and 4.11.

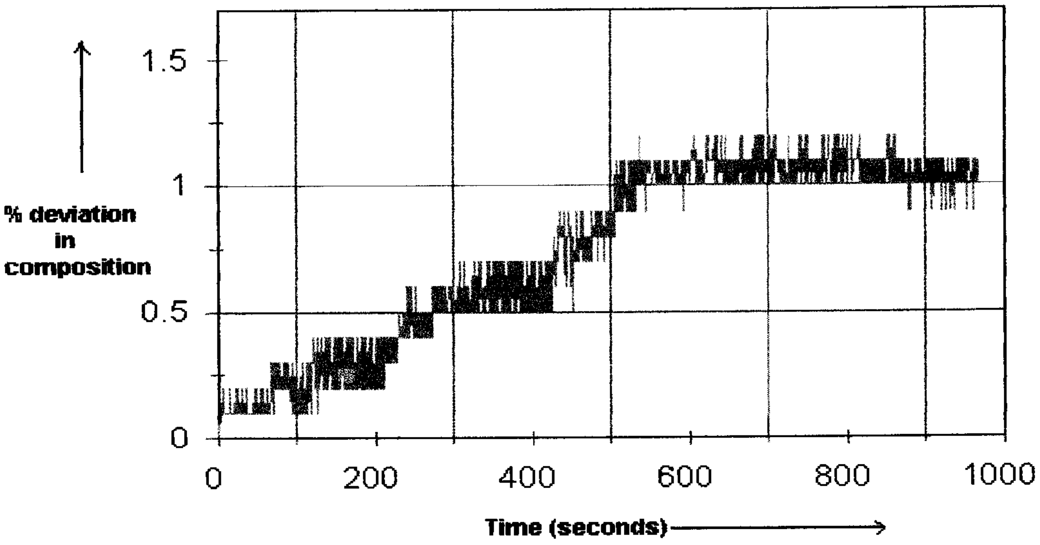


Figure 4.10. Closed loop Response of the column for 1 % change in the setpoint of the top product composition using the reflux valve as the final control element and capacitance measurements.

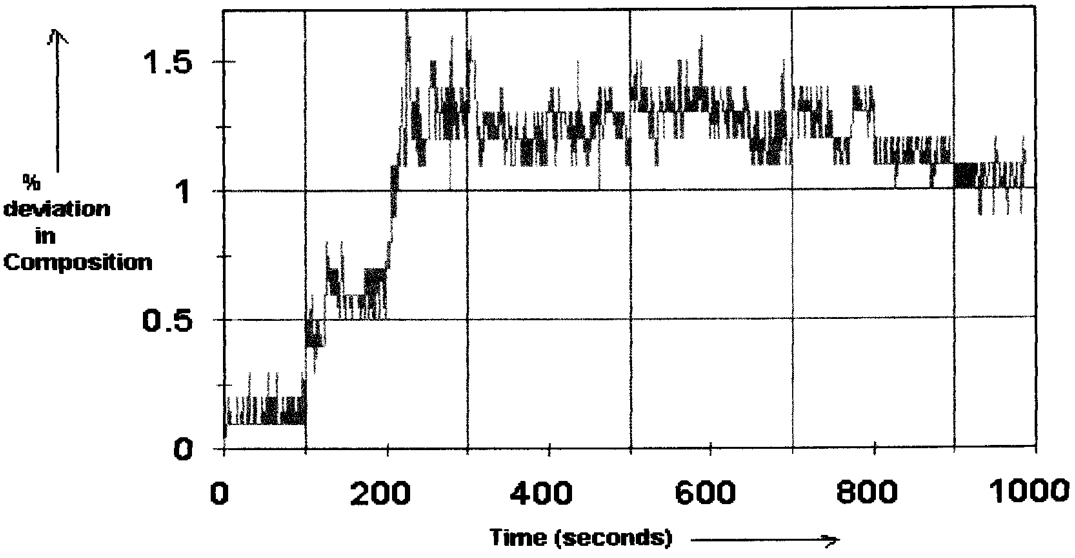


Figure 4.11. Closed loop Response of the column for 1 % change in the setpoint of the top product composition using the thyristor as the final control element and capacitance measurements.

UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

Chapter FIVE

Discussion of Results

The results presented in chapter four will be discussed with regard to the repeatability, accuracy, and reliability of measurements of the capacitance cell

5.1 Analysis of the capacitance cell readings

The capacitance cell was calibrated using compositions of 75%, 85%, and 90% ethanol and water. Each experiment was repeated 10 times with a particular composition, at three different temperatures namely 20°C, 30 °C, 40 °C. The mV readings obtained from 10 experiments were converted to composition using equation 4.2 and the data is presented in appendix I, Tables 1,2,3.

In order to ascertain the repeatability of the results obtained from the calibration process use of the theory of normal distribution was made. Figure 5.1 shows a typical normal distribution curve. According to this theory, there is about 70 % probability that data lies between μ-σ and μ +σ.

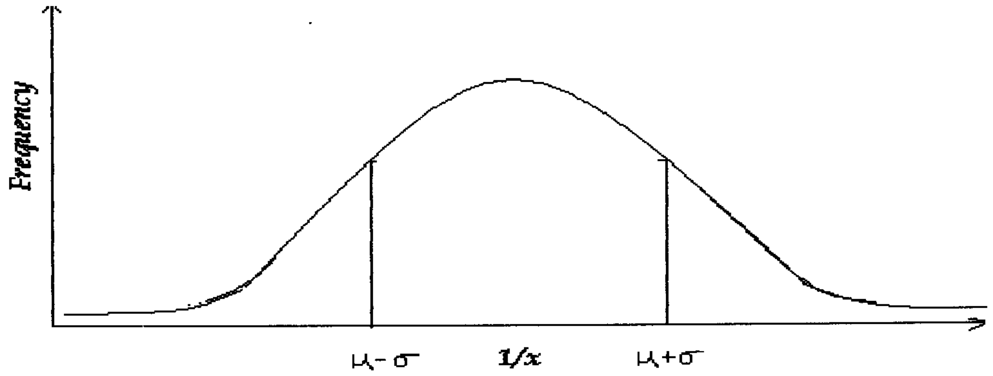


Figure 5.1 A Normal Distribution Curve

Similarly there is 95 % chance that data lies between $\mu-2\sigma$ and $\mu+2\sigma$ (Kirkup.L,1994). This method is very useful in analyzing experimental data, in as much as the smaller the standard deviation, the better the repeatability of a reading.

The calculated values of mean and standard deviation of the experimental data have been presented in Tables 4.1-4.3. It follows that for the three compositions 75%, 85% and 90% , at the three given temperatures the maximum value of standard deviation is 0,55 which is a good enough indication of repeatability for all practical purposes.

5.2. *Accuracy of the capacitance cell*

The capacitance cell should provide accurate readings that can be used for effective control of the column.

Therefore to check the accuracy, several changes in the reflux valve position were applied namely 0,25, 0,35 and 0,45 (fractional valve opening), from the steady state operating conditions of the column and values of composition were obtained for temperature, capacitance and density method. Figures 4.5-4.7 showed the results of these tests. From the data obtained for the reflux valve position of 0,45 for example and using equation 4.3, accuracy is calculated in respect of capacitance and temperature methods the details of which are given in appendix II.

The above stated tables show a statistically calculated mean and standard deviation of 98,57 and 0,795 respectively, for accuracy of the capacitance derived compositions, whereas the corresponding figures of the temperature derived composition is 98,121 and 1,21 respectively. The mean in the capacitance derived methods is closer to 100% accuracy of the density method, and also the standard deviation is smaller for capacitance derived composition compared to temperature methods, confirming that the capacitance method has given more accurate readings.

5.3. Dynamic responses of the column

To determine whether the measurement of composition via the capacitance cell had any negative influence upon the dynamic relation between top product composition and an input change, step disturbances in both the position of the reflux valve (magnitude of 0,15, fractional valve opening) and heat added to the reboiler, (magnitude of 50 %, thyristor heat input) were applied to determine the dynamic models for the column. Measurements with both temperature and capacitance techniques were used, and the response of the column for these disturbances was observed as shown in Figures 4.8(a,b,c)- 4.9 (a,b,c) and the process constants are given in Tables 4.5 and 4.6.

The two techniques provided nearly the same dynamic data. The temperature measurements were performed in the top plate of the distillation column due to reasons explained in section 2.4. and the capacitance cell measurements between the accumulator and the condenser. The capacitance cell provided an accurately enough reading at the point of measurement since in the installed position the reading which has already been shown, is accurate.

Models for both the measurement techniques are provided in equations 5.1 and 5.2 below. These models will not only determine the behaviour of the process ie response of top product composition over time but also will be useful in obtaining controller parameters in controlling the top product composition. In either case the sensing element is always in contact with the medium for which the composition is being measured.

Models obtained by temperature measurements:

$$\begin{pmatrix} y_L \\ y_Q \end{pmatrix} = \begin{bmatrix} \dfrac{28e^{-17s}}{55s+1} & 0 \\ 0 & \dfrac{-2{,}5e^{-12s}}{30s+1} \end{bmatrix} \begin{bmatrix} \text{Fractional valve opening} \\ \%\text{Thyristor input} \end{bmatrix} \tag{5.1}$$

Models obtained by capacitance measurements:

$$\begin{pmatrix} y_L \\ y_Q \end{pmatrix} = \begin{bmatrix} \dfrac{31.33e^{-17s}}{70s+1} & 0 \\ 0 & \dfrac{-2{,}9e^{-12s}}{35s+1} \end{bmatrix} \begin{bmatrix} \text{Fractional valve opening} \\ \%\text{Thyristor input} \end{bmatrix} \tag{5.2}$$

5.4 Performance of the capacitance cell under closed loop conditions

Closed loop control was performed on the column using the capacitance cell , in conjunction with the reflux valve and thyristor as final control elements to control top product composition. A PI controller was tuned and a 1 % change in setpoint of top product composition was applied. The response was monitored using the two final control elements in separate set of experiments, also given in Figures 4.10-4.11.

5.5 Dynamic lag

The dynamics of a measuring device plays a major role on the process, the larger the time constant or the time required for it to respond the slower is the controlling action. The dynamic lag was tested by five experiments in which composition of the feed to the capacitance cell was changed from 75% to 85 % ethanol. The response times for the five experiments were also given in Table 4.5. The largest response time was 1,3 seconds. The dynamic lag obtained corresponding to this value was 0,4 seconds, as shown in Figure 4.4 and is expressed by the transfer function 5.3

$$G(s) = \frac{1}{0{,}4s + 1} \qquad \textbf{(5.3)}$$

The effect that the dynamic lag has on the process is 0,4/70*100 = 0,57 (35 seconds is the time constant for reflux valve change) and 0,4/35*100 =1,4 % (70 seconds is the time constant for thyristor heat input change),which is negligible, showing that the capacitance cell does not significantly affect the process dynamics.

5.6 Ease of Operation

The capacitance measuring device is a cheap and simple piece of equipment, which is easy to fabricate, install and operate in conjunction with the electronic circuitry of which it forms part. It is also convenient for use on the column.

5.7Precautions and limitations

Effect of contamination :

There is a possibility of suspended particles in the flow stream to form a deposit on the capacitor plates which may cause variation from the original settings. This may be eliminated by incorporating suitable microfilters before the inlet to the capacitance cell. In case of temperature method the sensor is a thermocouple placed in the top tray of the column, and it is unlikely to be affected by contamination in the fluid.

Calibration:

Like in any instrumentation capacitance cell involves calibration. Although it appears lengthy during the initial stages, the process of calibration is easy and straightforward.

Dielectric property:

The capacitance cell can be used to determine composition of only dielectric fluids and cannot be used to determine the composition of reactive mixtures.

Multicomponent mixtures:

The capacitance cell can be used in determining composition of multi-component mixtures, provided that while compositions of two components vary, the composition of the other components should remain fixed.

Chapter SIX

Conclusions and Recommendations

This investigation was intended to develop and test a capacitance cell for top product composition measurement and control in a distillation column and to compare its performance with the presently used composition measurement techniques such as chromatographs, infrared analyzers and inferred measurements by temperature. Like in any other measurement technique it was necessary to observe the repeatability , accuracy and performance of the capacitance cell on column control.

The capacitance cell was tested for repeatable readings during the calibration procedure. Results obtained in a series of ten experiments were found to be normally distributed with a mean and the standard deviation as shown in Tables 4.1-4.3. The standard deviation values as observed from the tables were not greater than 0,55 which is a good enough indication for all practical purposes, confirming that the capacitance cell gave good repeatability.

Test results to determine the accuracy of capacitance and temperature methods on the column showed that the capacitance cell provided more accurate measurements of composition than those obtained via temperature method , suggesting its advantageous use on the column.

Open loop tests were conducted on the column to compare the measurements by temperature and capacitance and also to observe the influence the capacitance cell has on the relation between top product composition and an input change. These experiments showed that although the capacitance cell readings deviated from temperature measurements due to its positioning on the column, measurements via capacitance were found to be advantageous due to their direct and accurate measurements.

During the closed loop tests, it was observed that the capacitance cell proved to be effective in measuring and controlling the top product composition.

The capacitance cell can be used for measuring composition of any binary fluid mixture that has dielectric properties but cannot be used for measuring compositions of reactive mixtures. It can be used in determining composition of multi-component mixtures, provided that while compositions of two components vary the other components would remain fixed.

The capacitance cell is compact and portable which enables it to be very convenient to incorporate in the control system. The probability of having suspended particles in the flow stream to form a deposit on the capacitor plates is likely to cause variation of calibration values from the original settings. This may be eliminated by incorporating suitable microfilters before the inlet to the capacitance cell.

As is common practice in instrumentation the capacitance cell needs accurate calibration. Although it appears lengthy during the initial stages, the process of calibration is easy and straightforward and does not pose any problem.

While recommending improvements to the device it is suggested that the electronics on the instrument be redesigned to be able to compensate for ambient temperature changes. The improvements should take this into account and provide for temperature compensation of typically temperature dependent components such as transistors and oscillators that deviate under different ambient conditions.

The capacitance cell proved to be a useful device in measuring and controlling top product composition in distillation column, and makes a better choice than other methods. It also offers scope for further development and applications in industrial scale plants.

Appendix-I

Table 1 Readings obtained from 10 experiments at 20 ^{0}C.

C	Expt 1	Expt 2	Expt 3	Expt 4	Expt 5	Expt 6	Expt 7	Expt 8	Expt 9	Expt 10
75%	75,03	75,03	74,56	74,09	75,5	75,97	74,56	75,5	75,03	75,03
85%	84,9	84,43	84,9	85,37	85,37	84,43	84,9	85,37	84,9	84,43
90%	90,07	89,6	90,07	90,54	89,6	90,07	90,54	90,07	91,01	89,13

Table 2 Readings obtained from 10 experiments at 30 ^{0}C.

C	Expt 1	Expt 2	Expt 3	Expt 4	Expt 5	Expt 6	Expt 7	Expt 8	Expt 9	Expt 10
75%	75,22	75,69	75,22	74,75	75,69	74.75	75.22	74,28	75,22	76,16
85%	85,09	85,56	84,62	85,09	85,56	84,62	85,09	84,15	86,03	85,09
90%	89,79	90,73	90,26	88,85	89,32	89,79	89,79	89,32	90,26	89,79

Table 3 Readings obtained from 10 experiments at 40 ^{0}C.

C	Expt 1	Expt 2	Expt 3	Expt 4	Expt 5	Expt 6	Expt 7	Expt 8	Expt 9	Expt 10
75%	75,41	75,47	74,94	74,94	74,47	75,41	75,88	74	74,94	74,94
85%	84.81	84,34	85,75	85,28	84,87	85,75	85,28	84,81	84,34	84,87
90%	89,98	90,45	89,98	90,45	89,51	89,98	90,02	89,51	89,04	89,98

Appendix-II

Table 1. Sample data of 10 readings for a fractional change in reflux valve position of 0,45 by capacitance measurements.

Time (sec)	**0**	**50**	**100**	**150**	**200**	**250**	**300**	**350**	**400**	**450**	**500**
C(**capacit-ance)**	77,2	76.2	78,4	79,7	81	82,1	83,2	83,7	84,9	85,9	88,2
C(**density)**	76,2	76,2	77,6	79,4	80,3	81,4	81,8	82,4	83,2	83,8	85,8
Ac%	98,03	100	98,97	99,62	99,13	99,14	98,29	98,42	97,96	97,45	97,2

Table 2. Sample data of 10 readings for a fractional change in reflux valve position of 0,45 by temperature measurements.

Time (sec)	**0**	**50**	**100**	**150**	**200**	**250**	**300**	**350**	**400**	**450**	**500**
C(**temper-ature)**	76	76,1	75,3	76,9	77,6	79,2	79,3	81	82,2	82,5	85,3
C(**density)**	76,2	76,2	77,6	79,4	80,3	81,4	81,8	82,4	83,2	83,8	85,8
Ac%	99,73	99,86	97,03	96,85	96,63	97,3	98,95	98,3	98,8	98,45	99,41

Appendix-III

Commissioning and use of the capacitance cell on the column.

. Ensure that the computer and the interface between the computer and the column are switched off (Gericke,1992), (Van Niekerk , 1995).

. Ensure that

1. The liquid level in the heating unit is aligned with the heating element.
2. The liquid level in the supply drum is aligned with the heating element.

If not - Do not switch the trip switch of the heating element on at the control panel. First use the control/master/executive program and the pumps to bring the liquids to the desired levels.

. Ensure that - There is enough liquid in the storage drum. If not use the pressure release pipe on the top of the storage drum and plastic pipe to add more ethanol. Remember to periodically add water , because the water also evaporates from the system, but not at the same rate as the ethanol.

. Switch on all the switches on the control panel.

. Switch on thyristor and push the <START> button.

. Turn the air pressure supply tap on and then turn on the air pressure to the supply drum(NB-in this order and not the reverse)

. Turn on the cooling tap and do so until the tap stops making a noise.

. Switch the computer on.

. Begin the program BEHEER.

. Switch the interface on .

. Set the valves to 0. (use the F3 (valves) option on the menu of the program)

. Set the thyristor to 100 %.

. After 20 minutes the liquids on all the plates will start to bubble and the vapour will flow to the condenser drum.

. Lock the single loop controllers which control the levels in the reboiler and the reflux and the feedflowcontrol (use F1 (control loop) option on the menu of the program).

. Use the steady state operating conditions of the column (mentioned in Chapter four, Section 4.1).

. Wait for the system to reach steady state.(It is expected to take ±70 minutes)

Ensure that - The liquid circulates between the storage drum and the feed drum . if not, Switch the pump off after 30 seconds so that the air bubbles which have formed can be expelled.

Usage of Program

.The Program on the hard drive

.The control program BEHEER is in C:\DISTIL or C:\SRINU

.The data is stored in C:\DISTIL\DATASTOR.

.The control loop parameters can be adjusted by changing the file C:\DISTIL\SETUP.DAT

.The values of the composition filter can be altered by changing the file FILTER.DAT.

Application of the Capacitance cell

.The capacitance cell is placed between the accumulator and the condenser of the column.

.Readings of the capacitance cell can obtained as soon as the interface and the computer is switched on. A red light is observed on the top of the cell, indicating that it is ready for operation.

.Program on the hard drive that is used to obtain composition readings from the capacitance cell is c:\Srinu\capa.pas.

From the Turbo Pascal program procedure

. Take the mV readings from the capacitance cell. These readings are taken from channel 10 of the pc30 card.

.Takes the temperature readings in the flow stream through the capacitance cell. This is obtained from the thermocouple 3 of the pc73 card.

.Composition is obtained from the main program relation between the mV and temperature readings.

.Open loop tests are conducted using the capacitance cell as follows:

.Set up the column to its operating conditions,

.Settings of the reflux valve and the thyristor are changed in c:\Srinu\beheer.

.Composition readings are obtained from c:\srinu\capa.pas based on these changes.

.Closed loop control on the column using the capacitance cell is achieved as follows:

. For the reflux valve as the final control element the program C:\Srinu\capa1.pas is used.

This program consists of

.C:\Srinu\pc66.pas as a Turbo Pascal unit in which the reflux valve settings are specified.

.C:\Srinu\capa.pas as a Turbo Pascal unit in which composition readings from the capacitance cell are obtained.

A PI controller algorithm as a main program relating composition and reflux valve position. This program makes use of the above mentioned Turbo Pascal units. In it also given the controller parameters, ie controller gain and integral time

.From thyristor as the final control element the program C:\Srinu\capa2.pas is used.

This program consists of

.C:\Srinu\pc66.pas as the Turbo Pascal unit in which the thyristor settings are specified.

. C:\Srinu\capa.pas is used as a Turbo Pascal unit in which composition readings from the capacitance cell are obtained.

.A PI controller algorithm as a main program relating composition and thyristor. This program makes use of the above mentioned turbo pascal units. In it also given the controller parameters, ie controller gain and integral time

Filtering Data in Matlab

The following procedure is undertaken using Matlab to filter an unfiltered data.

. Run the program Matlab.

. load the unfiltered data from C:\Srinu\unfiltered.dat .

. Activate "Simulink" in Matlab.

. Three blocks are chosen in this program

.The first block uses is a "workspace" in which the unfiltered.dat is saved as unfiltered.mat

.The second block contains the "filter", where the filtering parameter α is appropriately adjusted.

.The third block is the output block in which the filtered values are obtained.

. The filtered data then obtained will be analyzed.

Shutdown procedure of the experimental apparatus

- Switch off all the switches on the control panel.
- Switch off thyristor and push the <STOP> button.
- Turn off the air pressure to the supply drum and turn off the air pressure supply tap .
- Turn off the cooling tap.
- Switch the interface off .
- Exit the program BEHEER.
- Switch the computer off.

List of References

1. Akerlof, G. (1932), " Dielectric constants of liquid mixtures", Journal of American Chemical Society , V 54 (11). pp - 4130-4131

2.Cheng, D. (1989) *Field and Wave Electromagnetics,* Addison - Wesley Publishing Company. pp 123-125

3. Coulson, J.M, Richardson, J.F & Sinnot, R.K (1983) *Chemical Engineering Design (Volume 6).* Pergomom.

4. Eagle Electric (1992) *User manual for PC73, PC30 , & PC66,* Cape Town, South Africa.

5. Gericke.J.(1992) " Die implementering en evaluering van 'n gevorgerde beheersisteem op 'n distillasieKolom ", M Eng dissertation, University of Pretoria.

6. *International Critical Tables of Numerical Data for PHYSICS , CHEMISTRY AND TECHNOLOGY* (1930), Vol VII, Mc- Graw Hill Company.

7.Jannsen, P.W.M. (1986), "Bilinear Identification of a distillation column",Ph.D. Thesis in Chemical & Process Engineering, University of Canterbury, Christchurch, New Zealand.

8. Kip, A. F. (1965) *Fundamentals of Electricity and Magnetism,* Second Edition, Mc Graw Hill, New York. pp 47-48

9. Kirkup,L.(1994) *Experimental Methods: An introduction to the analysis and*

presentation of data, Wiley.pp 55-101.

10. Hougen, O.A , Watson, K.M, Ragatz, R.A (1958) *Chemical Process Principles*, John Wiley and sons, Inc.pp 56- 59

11. Horowitz ,M. (1989) , *The Art of Electronics*, Second edition. W Cambridge University Press. pp 297-298, pp 231-232.

12. Manahan, S.E. (1986) *Quantitative Chemical Analysis*, Brookes/Cole publishing company.

13. Meehan, E.J. (1966) *Optical method of Analysis*, InterScience Publishers.

14. Olsen, E.D. (1975) *Modern Optical methods of Analysis*, Mc-Graw Hill.

15. Pecksok, R.C. & Shields L.D. (1986) *Modern Methods of Chemical Analysis*, John Wiley and Sons.

16. Poole, C.F &Poole, S.K. (1991) *Chromatography Today*, Elsevier Science Publishers.

17. Rhea, R.E. (1990), *Oscillator Design and Computer Simulation*, Prentice - Hall, INC. pp 23-155

18. Perry, R.H & Green, G. (1987) *Chemical Engineers Handbook*, Mc-Graw-Hill International.

19. Rys,R.A. (1984), " Advanced Control Techniques for Distillation Columns",

Chemical Engineering, December, pp- 75-81.

20. Sandler,S.I. (1989) *Chemical and Engineering thermodynamics,* Second edition, Wiley series in chemical engineering, pp 321-337.

21. Schomburg,G. (1990) *Gas Chromatography, A Practical Course,* VCH.pp 5-7

22. Shinskey, F.G.(1988) *Process control systems* - Application design and tuning, Mc- Graw Hill Inc. pp 431-445

23. Smith, J.M. and Van Ness. H.C. (1987) *Introduction to Chemical Engineering thermodynamics,* fourth edition, Mc Graw Hill, New York.

24. Stanley, B. H. (1985) *Experiments in Electric Circuits,* Second edition, Bell and Howell Company. pp 129-180

25. Stanton, B.D.(1986)" Designing Front-end control schemes for distillation towers" ; Chemical Engineering, Vol.24,November ,pp. 87-92.

26. Stephanopoulos.G (1984) *Chemical Process control, An introduction to theory and practise,* Prentice-Hall International, Inc.

27. Svrceck,W.Y. (1967) "Dynamic response of a binary distillation column ", PhD Thesis, Department of Chemical and Petroleum Engineering , University of Alberta, Edmonton. pp 57-67.

28. Swan ,T.(1988), *Mastering Turbo Pascal 4.0*, Second edition, Hayden Books.

29. Van Niekerk . F.J. (1995) "Die implementering van 'n Multiverranderlike internemodelbeheerer op 'n distillasiekolom", MEng dissertation, University of Pretoria.

30. Walker ,C.S.(1990) *Capacitance, Inductance, Cross Talk Analysis*, Artech House. pp15-17

31. Young, J. F. (1968), *Applied Electronics*, London Ileffe Books Ltd. pp 73-107