# STUDY ON EFFECT OF WITHERING RATE AND QUALITY OF

### **MADE TEA**

at



Tea Research Institute- Talawakelle

By

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A research report submitted in Partial Fulfilment of the Requirements of the Food Science and Technology

of

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#### **DECLARATION**

I carried out the work described in this thesis at Faculty of Applied Sciences and Tea Research Institute (TRI) under the supervision of Mrs. Rasangi Sabaragamuwa and Mr. K. Raveendran. A report on this has not been submitted to any other university for another degree.

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# AFFECTIONATELY

# **DEDICATED**

# TO

# **MY PARENTS**

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I express my sincere thanks Mrs. Rasangi Sabaragamuwa , Lecturer, Faculty of Applied Sciences, Sabaragamuwa University of Sri Lanka, Buttala for their valuable assistance towards for the successful completion of the training programme.

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I also express my thanks to TRI staffs and other workers for their moral support given during the entire period of the research project.

#### ABSTRACT

Withering is the one of the basic step in manufacturing process of tea. To obtain good quality tea the withering rate should be maintained at the optimum rate. A study was carried out to find out the effect of withering rate on quality of made tea, at St. Coombs Estate, Talawakelle managed by the Tea Research Institute.

Tealeaves were obtained from a three-year-old vegetatively propergated (VP) tea field. Two tea cultivars were selected, DT 1 of high quality and TRI 2025 of poor quality. This experiment was carried out to study the affects of withering rate on quality of made tea by applying different hygrometry differences ranges between 4- 8  $^{\circ}$  F. Different withering rates were achieved by changing hygrometry differences. 5  $^{\circ}$  F, 6  $^{\circ}$  F, 7  $^{\circ}$  F and 8  $^{\circ}$  F are the treatments and 4  $^{\circ}$  F considered as control in a Randomized complete block design (RCBD). The time allocated for plucking was from 10.30 a.m to 12.30 p.m with six days plucking interval for each block.

The samples were manufactured using miniature tea machinery and the made tea samples chemically evaluated using Robert & Smith method (1962). The level of TF, TR, TF: TR, Total colour and brightness of liquor were determined as quality parameters by this method. The results obtained were significant (P=0.05) for DT1 than TRI 2025. The final result showed a significance difference in quality of made tea of DT1 cultivar at 5° F and of TRI 2025 cultivar at 6° F. From this result it can be concluded that to obtain good quality made tea the hygrometry difference should be selected properly. According to this research study there is a direct relationship between withering rate and level of quality parameters.

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# **ABBREVIATIONS**

| °C.    | Degrees of Centigrade             |
|--------|-----------------------------------|
| ° F .  | Degrees of Feranide               |
| H.D    | Hygrometry Difference             |
| BM     | Broken Mixed                      |
| BOP    | Broken Orange Pekoe               |
| BOPF · | Broken Orange Pekoe Fanning       |
| BPF    | Broken Pekoe Fanning              |
| BP     | Broken Pekoe                      |
| cfm    | Cubic feet per minute             |
| CTC    | Crush tear curl                   |
| DT 1   | Drayton                           |
| ECM    | Environmental Control Manufacture |
| FBOP   | Flowery Broken Orange Pekoe       |
| FBOPF  | Flowery Broken Orange Pekoe       |
| FP     | Flowery Pekoe                     |
| g .    | grams                             |
| GL     | Green Leaf                        |
| Kg     | Kilograms                         |
| min ·  | minute                            |
| OP     | Orange Pekoe                      |
| Р      | Pekoe                             |
| PF     | Pekoe Fanning                     |
| rpm    | Revolution per minute             |
|        |                                   |

# CHAPTER 01 1. INTRODUCTION

#### 1.1 History of tea

Tea is the cheapest hot beverage consumed by over two thirds of world's population. The leading tea producing countries are located in tropical and subtropical regions such as China, Japan, Taiwan, Indonesia, India and Sri Lanka. It ranks as number one foreign exchange earner for the country. This crop successfully replaced Coffee that was totally devastated by the leaf blight disease (*Hemileia vastatrix*). Tea was introduced to the country in 1839 about 30 years prior to the occurrence of the coffee disease in 1869. The first commercial planting in Sri Lanka was introduced by James Taylor and cultivated at 19 acres in Loolkandura Estate at Hewahata. Tea is a drink made by infusing leaves of the tea plant (*Camellia sinensis*, or *Thea sinensis*) in hot water. (Robert, 1986) The three main categories are green, black, and oolong. All three kinds are made from the same plant species. The major differences between them are a result of the different processing methods they undergo. Black teas undergo several hours of oxidation in their preparation for market; oolong receive less oxidation, and green teas are not oxidized at all.

#### 1.2 Botany of tea

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Tea is most important of all Camellia species both commercially and taxonomically. The genus Camellia includes 82 species, which are mostly indigenous to highlands of South-East Asia (Sealy, 1958). Tea was classified by Linnaeus (1752) as *Thea sinensis*. The recognized two varieties are *Camellia sinensis* var. *sinensis*(L.), covering all narrow leaved China types of plants and *Camellia sinensis* var. *assamica*(Masters) covering the broad leaves Assam type of plants. The Assam variety was first described by Masters (1844) as *Thea assamica*, and the two major varieties of tea were also distinguished by Kitamura (1950) as *Camellia sinensis* var. *assamica* (Masters) and var. *sinensis* (L). He further classified a third type of tea plant and included under C. *assamica* as a sub species. This type is referred as "Cambodia race" or "southern form of tea". Although tea varieties differ markedly in their growth rate and size and the shape, size, texture and geometry of their leaves.

#### 1.3 Requirements for tea cultivation

Tea grows from 2000m from height from sea level and the annual rainfall should be1500-5300mm. Environmental minimum and maximum temperature should be 14°C and 28°C respectively. Tea grows well between pH 4.5 and 5.6 of soil and Nitrogen content of the soil should be 0.8-0.9%. Hard or stiff subsoil, depsite satisfactory topsoil, affects the growth and development of roots. Also the soil should be deep, porous and friable to a good depth. (Watson, 1986)

#### 1.4 Biochemistry of Tea

Tealeaves contain mainly Polyphenolic compounds, enzymes, Amino acids, carbohydrates, lipids, chlorophyll and carotenoids, volatile compounds, vitamins and minerals. The best tea comes from the tiny young shoots and the thin unopened buds of the flowering evergreen plant. All tea is derived from same plant species but differs in types and variety according to the region in which it is grown, the manner in which it is plucked and finally the way it is manufactured. By exposing the tea leaves to varying degrees of evaporation, twisting, oxidation and heat, the three great tea classifications are made. They are Black, Green and Oolong tea. From the plucking to processing, grading, packing, the proper conditions should be maintained for a good quality tea.

| Components                  | Concentrations (g/100g) |
|-----------------------------|-------------------------|
| Catechins                   | 3                       |
| Theaflavins                 | 3                       |
| Thearubigens                | 12                      |
| Flavanols                   | 6                       |
| Phenolic acids and depsides | 10                      |
| Amino acids                 | 13                      |
| MethylXanthines             | 8                       |
| Carbohydrates               | 10                      |
| Proteins                    | 0.8                     |
| Mineral matter              | 8                       |
| Volatiles                   | 0.05                    |

Table 1.1 Principal components of black tea beverage

The main attributes of tea consumers expect its taste, aroma, strength and freshness of the brew. For a good quality black tea has been found to be associated with the levels of Theaflavin (TF) and Thearubigen (TR) and highly polymerized substances as well as ratio between them. When the manufacturing process takes place a series of physical and/or chemical changes, transforming them into one of the three major black, green and oolong tea. Black tea must be withered, rolled, broken, fermented and fired.

#### 1.5 Wither

Withering is the first step of processing in the factory and is a process in which freshly plucked leaf is conditioned physically as well as chemically for subsequent processing stages. Based on achieving desired level of withering, can make better quality of tea and on neglect, can cause serious problems in subsequent steps of manufacture. The process objectives achieved during withering as follows;

- Reduce moisture content of fresh leaves ranges between 70-83%.
- > Changes in enzyme activity
- Partial breakdown of proteins to amino acids which act as precursor for aroma.
- Increase in caffeine content.
- Reduction in chlorophyll content.

The withering time takes about 10-18 hours. The degree of wither is determined by the percentage of moisture removal in a defined period of time, to a specified hygrometry difference between the wet and dry bulb temperature. Above chemical changes are all intrinsic of the biochemical structure of leaf, and the extent of the reactions depends on cultural practices and physical parameters such as temperature, humidity etc.

#### 1.6 Hygrometry difference

Dry and wet bulb thermometers are used to measure humidity of withering air. The difference between dry and wet bulb temperature means the hygrometry difference (H.D). The TF, TR, totalcolour and brightness the effect of H.D on tea quality in different varieties can be achieved.

# 1.7 Objectives

## **Main objectives**

- 1. Study the effect of withering rate on quality of made tea.
- 2. Study the effect of withering rate in high quality clone DT1 and poor quality clone TRI 2025.

### **Specific objectives**

1.Optimization of withering process by obtaining a specific hygrometry difference by studying the biochemical quality parameters.

### CHAPTER 02

## **2. LITERATURE REVIEW**

#### 2.1 Raw materials

Quality of the end product is mainly depending on the raw material. The raw material of the tea is the green leaves. The plucking standard, age of bush, stage from previous pruning, plucking round etc. are some of the important factors that contribute to the quality of the tea shoots. Besides, in the same shoot, the chemical composition of 1st leaf, 2nd leaf, other leaves, stem, etc. vary widely.

#### 2.1.2 Propagation

Tea can be Propagated either by Seed or by vegetative means.

#### 2.1.2.1 Clonal Tea

Plants produced vegetatively from a single parent plant is referred to as clone. The yield potential of clonal tea in early trial was so impressive therefore Government of Ceylon introduced legislation in late 1958 to make it compulsory to use approved clones for the tea estates in their new clearings. Main advantage of using clone is they have uniformity among the progeny. Any other variability within a clone will be due to the effect of environment. Other advantages are high yield, good quality, ease of vegetative propagation, tolerance to pest and diseases.

#### 2.1.2.2 Approved Clones

1

Initially there were over 200 approved clones, but now only about 40 were being used, of these only about 10-15 are popularly used and over 95% of the replanted clones represent TRI selection (mostly TRI 2020 series), while others constitute estate selections.

- The two most popular clones in the low country were TRI 2023 and TRI 2026. In recent years TRI 2025 becomes popular due to it's hardness and tolerance to drought and eelworm, high yield potential and vigour of growth.

TRI 2043 red-pigmented clone, which has buds, covered with silvery white hairs is popular on some low country estates because it produces tippy teas for which there is a big demand in Middle East market.

5

Other clones selected by estates are Sirikandura(S) 106; Karapincha (KP) 204 and Ragalla (B) 275 have high yield potential. Drayton (DT) 1 is of high quality; Balangoda (DG) 7fairly resistant to drought and Drayton (DT) 95, Kirkoswald (K) 145 and Norwood (N)2 are highly tolerant to eelworm.TRI 3013,3014,3015,3016,3017,3018,3019 and 3020 are recommended for small scale planting.

#### 2.2 Pruning

Pruning is an artificial removal of leaf bearing branches of the plant. Aim of this pruning is keeping the size and vegetative vigor of the plant in a condition most conducive for maximum vegetative growth and cropping. Pruning stimulates and controls growth.

Main Objectives of Pruning are;

1. Shaping the tree to make the best use of space between trees.

2. Stimulation of vegetative shoot growth.

3. Maintenance of a healthy frame.

4. Maximize crop production but minimizes the spread of pests and diseases.

5.maintaining quality of made tea.

Plant variety, environmental factors and general growing conditions affect the measure necessary to maintain a particular pruning system.

#### 2.3 Green leaf standard

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Green leaf is the raw material for black tea processing and it is important to have a good quality black tea. Good leaf is named as "Tender leaf". 100% good leaf is not practicable. Therefore TRI recommends 65% good leaf (on count basis) as acceptable (Tubb 1949). The liquoring quality of a tea is measured by the amount of catechins or primary polyphenols and caffeine. If the plucking is coarse, the percentage weight of two-leaf shoot will be much less. An average plucking standard should have ideally about 75% fine leaf by weight comprising bud and two leaves, soft banji, undeveloped bud plus two leaves. For achieving quality the fine leaf should not be diluted with coarser leaf. Approximate contribution of different shoot components in different plucking standards is given by the below table.

6

| шy | as 10110 ws.    |                  |                  |
|----|-----------------|------------------|------------------|
|    | Shoot component | % in 2 and a bud | % in 3 and a bud |
|    | Bud             | 14.0             | 7.0              |
|    | 1st leaf        | 24.0             | 12.0             |
|    | 2nd leaf        | 45.0             | 24.0             |
|    | 3rd leaf        | -                | 33.0             |
|    | Upper stem      | 17.0             | 8.5              |
|    | Lower stem      | -                | 15.5             |
|    |                 |                  |                  |

Depending on the plucking standard, the tender components of the shoots vary as follows:

Table 2.1 Components of tea shoots vary with plucking standard

The figures given above may vary because of climatic and other factors, but are fairly representative.

#### 2.4 Plucking

Plucking denotes harvesting of tea crop by selective hand picking of tender shoots called as plucking. The shoots are plucked at the interval of 4-10 days. The crop shoot consists terminal bud and the internodes (stalks) and 1, 2 and three leaves immediately from the bud.(Tubb, 1949) The objective of plucking is to harvest the maximum yield of good quality leaf per unit area, combined with maximum labour efficiency. Plucking standard describes as fine, medium or coarse depending on the size and maturity of the shoots taken.

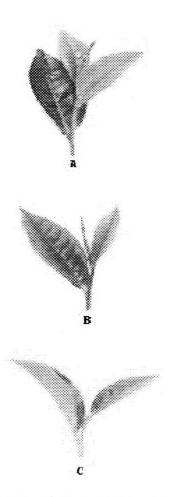
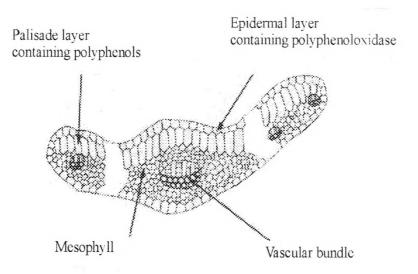


Fig 2.2. **A** - Three leaves and a bud **B** - Two leaves and a bud **C** - Banji shoot

## 2.5 Tea Biochemistry

Biochemical characteristics are the basic requirements for the different phases of processing. Tea contains enzymes, amino acids, phosphate esters, nucleotides and caffeine, carbohydrates, lipids, chlorophyll, and carotenoids, volatile compounds.Tealeaf contains polyphenolic compounds, polyphenol oxidase enzyme, etc.



#### Fig 2.2 Schematic diagram of a tealeaf

#### 2.6 Components of black tea

#### 2.6.1 Enzymes

The enzymes present in Tea leaves are;

#### Leaf polyphenol Oxidase(PPO)

Tealeaf polyphenol oxidase (o-diphenol  $:O_2$  oxidoreductase 1.10.3.1) important in processing of black tea. PPO has ability of reacting with the polyphenols present in the leaf. The concentration of chemical compounds in younger leaves is ideal for the production of good quality. Coarser leaves and stems contain lower concentrations of enzymes as well as other components.

#### 2,5- Dehydroshikimate Reductase

Activity of this enzyme is significant in growing shoots than in mature leaves and is crucial in the biosynthesis of polyphenols via phenyl alanine pathway (Sanderson, 1966).Other enzymes present in tea leaves are Leaf Peroxidase, Leaf Peptidase, Leaf Chlorophyllase, Acid Phosphatases, Leucine-alpha-ketoglutarate Transaminase,Alcohol dehydrogenase,Pectin Methyl Esterase and Leaf Ribonuclease.

#### 2.6.2 Polyphenolic compounds

The most important characteristic component of tea leaves are the polyphenolic compounds, present in the cytoplasmic vacuoles of tea leaf cells.Flavanols are oxidized by polyphenol oxidase enzyme and it produces compounds that determinates the colour of the tea brew. Most have chemical configuration based on Catechin and Gallic Acid.

Polyphenolic oxidase
Polyphenol \_\_\_\_\_ The

Theaflavin+Thearubigen

Polyphenols in tea flush are;

a) Flavanols

b) Flavanols and Flavanol glycosides

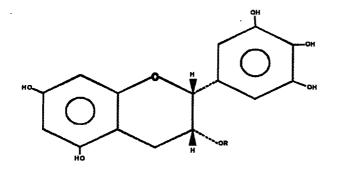
c) Flavones

d) Acids and Depsides

(a)Flavanols

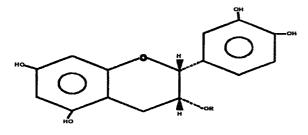
Belongs to class of catechin 25-30% of drymatter of tealeaves. Soluble in water, astringent taste and colourless.

Flavanols present in tea are shown below. (Fig 2.3)



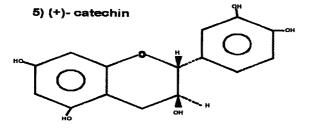
 1)(-) –epigallocatechin gallate R=3,4,5-trihydroxybenzoyl
 2)(-) \_epigallocatechin

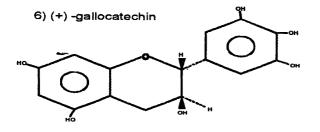
R=H

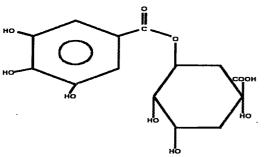


- 3) (-) –epicatechin gallate R=3,4,5-trihydroxybenzoyl
- 4) (-) epicatechin

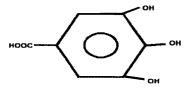
R=H



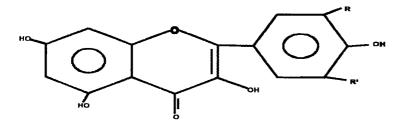








(8) GALLICACID



(9) FLAVONOLS

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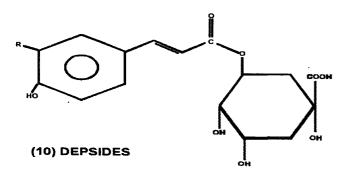


Fig. 2.3 Flavanols present in tea

#### 2.6.3 Aminoacids

The proteins in tea do not play a significant nutritional role but aminoacids are the responsible compounds for aroma formation. However ,Theanine (5-Nethylglutamine)-HOOC.CH (NH<sub>2</sub>).CH<sub>2</sub>CH<sub>2</sub>CO.NH.C<sub>2</sub>H<sub>5</sub>, is an aminoacid unique to tea.The other aminoacids in tea leaf are, aspartic,glycine,alanine,valine,leucine,isoleucine,methionine and phenyl alanine are commonly found in other plants as well these aminoacids play a significant role in aroma formation the total protein intake from tea alone is unlikely to be more than 70mg/day.Asparagine formed during the withering process.

Volatile carbonyl compounds formed from the proteins during processing;

| Glycine       |   | formaldehyde       |
|---------------|---|--------------------|
| Alanine       |   | Acetadehyde        |
| Valine        |   | isobutryldehyde    |
| Leucine       | > | isovaleradehyde    |
| Methinine     |   | 2-methylbutanol    |
| Methionine    |   | methional          |
| Phenylalanine |   | phenylacetaldehyde |

#### 2.6.4 Lipids

Tea consists of phospholipids, glycolipids, sulpholipid and triglycerides.the unsaturated fatty acids viz. linoleic and linolenic acids derived from the phospholipids contribute to the aroma formation.

#### 2.6.5 Caffeine

1

Caffeine (1,3,7-trimethyl xanthine) is present in 3-5% in drymatter of tea leaf. It is a purine derivative. It has stimulating property and removes mental fatigue. The contribution of caffeine to the infusion is the briskness and creamy property resulting from the complex formed by the caffeine with polyphenols.

#### 2.6.6 Tea fibre

The leaf cell wall, containing cellulostic materials surrounded by hemicellulose and lignin seal prevents the penetration of hydrolyzing enzymes. The reduced succulence in the matured shoot is believed to be due to structural bonding between phenolic components of lignin, polysaccharides and cutin of cell wall.

#### 2.6.7 Carbohydrates

The free sugars present in tea shoot are glucose, fructose, sucrose, raffinose and stachylose. Maltose in Assaam variety and rhamnose in china variety appeared special. Pectic substances contain galactose, arabinose, galactouranic acids. Formation of heterocyclic flavor compounds during processing of black tea and contributing towards water soluble solids in tea liquor.

#### 2.6.8 Carotenoids and Chlorophyll

The four major carotenoids a Present in tea leaves are  $\beta$ carotene,lutein,violaxanthine and neoxanthine. They vary with plant types and processing of tea leaves.

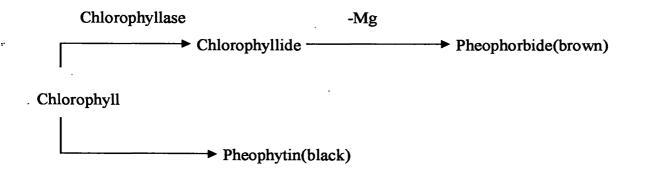


Fig 2.4 Changes in Chlorophyll during processing of black tea. (Wickramasinghe, 1978)

#### 2.7 Manufacturing of tea

The Black Tea processing contains four basic steps. They are withering, rolling, fermentation, drying, grading and packing (Keegel, 1983).

#### 2.7.1 Withering

Withering of tealeaves involves the extraction of part of the moisture in green leaf under controlled condition using a mixture of hot air and ambient air. Withering reduces green leaf of moisture 70-83% to 55-60%. In this withering process the green leaves reduced from it's turgid condition to a pliable form, which is suitable for next processing stage, which is the rolling by the uniform and gradual loss of moisture.

2.7.1.1 Expressing wither

Wither can be expressed in two ways.

- Percentage of wither -not common in Sri Lanka.
- Degree of wither More relevant to the quality of tea produced.

#### **Percentage wither**

The ratio of out turn withered leaf to green leaf expressed as a percentage.

Withered leaf (Kg) \_\_\_\_ Percentage wither **X** 100 Green leaf (Kg)

#### **Degree of wither**

The ratio of out turn made tea to withered leaf expressed as a percentage.

Made tea(Kg) Degree of wither = X 100

Withered leaf (Kg)

The out turn MT/WL and moisture content of withered leaf can be expressed approximately as:

#### % Out-turn MT/WL =100-% moisture content of withered leaf on wet basis

The correct or most appropriate way to check the degree of wither is to check the moisture content in withered leaf.

The microwave oven method and standard oven method could be used to predict the wither accurately. (Mohamed, 1998)

The degree of wither aimed should mainly suit the type of manufacturing system.

| Low-country manufacture |               | Pure orthodox system -41-42%      |
|-------------------------|---------------|-----------------------------------|
| MT/WL                   |               |                                   |
| Up country manufacture  | $\rightarrow$ | pure orthodox system-43-45%       |
| MT/WL                   |               |                                   |
|                         |               | Orthodox-rotor vane system-43-45% |
| MT/WL                   |               |                                   |
| CTC manufacture         |               | 30% MT/WL                         |

The degree of wither is referred to as percentage of moisture extracted by its weight from the green leaf and can vary from 25-50% as indicated below depending on the type of processing.

| Manufacturing type | % Moisture  | Wither    |
|--------------------|-------------|-----------|
|                    | content wet | type      |
|                    | basis       |           |
| CTC/LTP            | 70          | very soft |
| Orthodox           | 58          | soft      |
| Orthodox/Rotorvane | 55          | Medium    |
| Rotorvane          | 52          | Hard      |

Table 2.2 Types of withering

During wet months it is necessary to use more hot air to remove surface moisture, and then regulate both the hot and ambient air to ensure an even wither thereafter. Thus a considerable supervision is necessary at initial stage.

The degree of wither is generally determined with the experience by touching, smell. But the most accurate method is reweighing a predetermined weight of green leaf after withering and expresses same as a % of moisture extracted.

#### 2.7.1.2 Trough withering

In factories they use trough that has dimensions as  $10^{1/12}$  deep sections, 6 ft wide,  $3\frac{1}{2}/1\frac{1}{2}$  ft. fitted with axial flow fan units, complete with hot and ambient air controls and tapered transformation ducts.

The capacity of trough unit depends on size of fan and motor required to deliver the airflow needed for the accepted spread of Greenleaf at static pressure.Generally using maximum possible trough length for the generally available size of fans and motors based on the following;

Maximum spread of Green leaf -7 lb/3.2 Kg per sq ft

Airflow per Kg of green Leaf -20-c.f.m

Static Pressure - 0.6"/15 mmHg

Due to varying distances between heat source and each trough unit it is necessary to mix hot air and ambient air and expose green leaf should withered at same temperature and humidity. The uniformity of wither is absolutely essential (staff, 1971).

To ensure efficient withering to maximum and minimum hygrometry difference should be 4°F, 8°F respectively. Hygrometry difference can be monitored with the use of dry bulb and wet bulb thermometers. Withering process takes from 8-16 hrs depending on the type of manufacture and other condition.

#### 2.7.1.3 Physical Wither

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Physical withering process consists;

-To reduce the moisture content of the fresh leaf, ranges between 74-83%.

-To make leaf "flaccid" or "Rubbery" this is essential for the next step of maceration or rather for "twisting' or curling, etc.

Physical wither can be achieved within 3-4 hrs. But chemical wither requires 12-16 hrs.

#### 2.7.1.4 Chemical wither

Chemical wither starts immediately after plucking. It is independent of the rate of the moisture and is a function of time and temperature. Although the desired moisture level may be reached in a few hours, the catabolic changes, which had been initiated at the time of plucking, will take time. Longer period is need for chemical wither. The following chemical changes occur during withering;

-Release of Carbon dioxide and water due to break down of larger molecules.

-Changes in enzyme activity.

- Partial breakdown of proteins to amino acids which act as precursors for aroma.

-Increase in caffeine content-contributes to briskness.

-Production of volatile flavor compounds. (VFC)

-Reduction of Chlorophyll content.

These reactions depend on time and temperature, humidity, Cultural practices and withering methods. The various withering methods are tunnel withering, Drum withering, and trough withering has been in use. Generally tea factories use trough withering. Because;

-Economy

-Greater flexibility with respect to capacity of degree of wither

-Flexibility of construction

-Saving in space.

-Economy of labour and easier operation.

#### 2.7.2 Rolling

Rolling may defined as rupturing of cells in the withered leaf by mechanical means resulting in reduction of particle size, release of enzyme and initiation of the fermentation process. Most widely used rolling types are;

1. Orthodox roller - batch process

2. Rotorvane - Continuous process

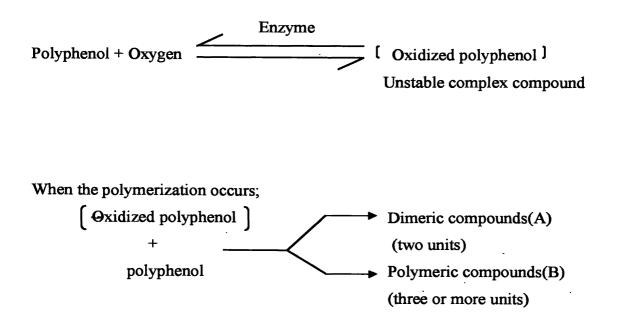
#### 3. C.T.C (Crush,tear.curl) - Continuous process

#### 4. LTP (Lawrie tea processor)- Continuous process

The finer particles of tealeaves called as "Dhools" being extracted after each roll by means of rotating or reciprocating sifters normally referred to as roll breakers. The larger particles remains on the roll breaker is called "Big Bulk". During the rolling, certain amount of heat generated, which can effect the quality of tea, thus the pressure is released at specific interval during the rolling, to provide some aeration that maintain temperature within an acceptable level about 85oF/30oC. Then the leaves are twisted or curled. Additionally, the rolled leaves can be processed by a machine: its sharp teeth cut the leaf then tear it to release the juices that contain the tea flavor, and intensifies the fermentation process.

#### 2.7.3 Fermentation

Fermentation is a complex chemical reaction that begins at the moment of leaf is broken in the roller. The breaking of cells causing the mixing of the enzymes with the other chemical components within the cell results in a number of reactions. The most important reaction is conversion of polyphenols.



The result of above reaction is orange red Theaflavin (TF) and compound and darker brown Thearubigen (TR) compounds can be produced. TF is dimeric compound and the TR can contain predominantly polymeric units. Hence some dimeric units also present.

The quantity of TF and TR formed will be related to the fermentation as well as to the temperature of the fermenting leaf. TR rapidly increases with the high temperature, which will impart dull colour of the liquor. The standard fermentation time should be 2-  $2\frac{1}{2}$  hours from the production of first dhool production. At the ratio of TF: TR range should be 1:10 to 1:12 for the idealized liquor colour and brightness(Basu ullah, 1978).But it can vary with the type of tea, the rolling programme, season and temperature.

The thickness should be 3 inches of dhools and the hygrometry difference (Relative humidity) in the area should be 1-3  $^{\circ}F$ .

Too low R.H - " Surface drying" of dhools occur.

Too high R.H - " condensation of water on the particles and retard the fermenting rate.

At the tea factories broken leaf is laid out, either on trays or in troughs, in a cool, humid atmosphere for up to two hours to ferment, or more correctly, oxidize. The trays are gently turned every so often throughout the period until all the leaves turn a golden russet color and fermentation is complete.

#### 2.7.4 Drying

Drying removes most of the moisture present in the Dhools.Final fired dhool's moisture content must be 3-4%(Evans, 1932). To prevent the activity of polyphenol oxidase enzyme, the firing has to be done. Standard temperature and time combination should be 160 ° C-210 ° C for 21 minutes. Endless chain pressure (ECP) and fluidized bed dryer (FBD) are most common in SriLanka.(Mohamed, 2000)

| Dryer | Inlet      | Exhaust   |
|-------|------------|-----------|
| FBD   | 127-129° C | 88-93 ° C |
| ECP   | 88-90 ° C  | 52-54 ° C |

Table 2.2 Temperature limitations for common dryers

#### 2.7.5 Grading

Graded tea should confirm to accepted criteria on particle sizes, particle shapes, heaviness, evenness, and cleanliness. Grading achieved by;

-Dividing in to fractions according to particle size

-Cleaning of tea from stalk and fibre

-Reducing particle sizes of oversize particles

Chota and miche sifters are the most common tea grading machineries in SriLanka.

Main grades are;

O.P – Orange Pekoe

F.B.O.P – Flowery Orange Pekoe

B.O.P.1 – Broken Orange Pekoe No. 1

F.B.O.P.F - Flowery Broken Orange Pekoe fannings

B.O.P - Broken Orange Pekoe

B.O.P.F- Broken Orange Pekoe Fannings

Off grades are; B.P - Broken Pekoe B.M - Broken Mixed B.M.F - Broken Mixed Fannings

Dust No. 1

Dust No.2

#### 2.7.6 Packing

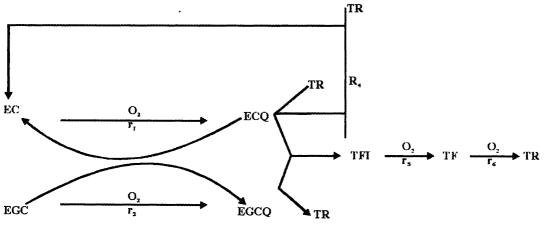
After the firing of tea, tea should be stored in large bins prior to packing. These bins are constructed by timber and lined with aluminium sheeting prevent the absorption of moisture.

#### 2.8 Quality of made tea

Quality of made tea is determined by the taste, colour and the aroma of made tea.

### 2.8.1Aroma

Tea aroma is composed of various organic compounds and said to be one of the important factor to determine the quality of each made tea. The contributors for the aroma formation are, glycosidic aroma precursors of alcoholic tea aroma such as geraniol, linalool, 2-methyl salicilate, linalool oxides, etc. Which contributes to the floral aroma of Oolong tea (semi fermented) and black tea (fermented tea) as well as enzyme concerned with the alcoholic aroma formation from the aroma precursors. (Yamanishi, 1989b)



(Robertson, 1983 a)

Fig 2.5 Scheme showing the formation of theaflavin and thearubigen from the catechins and possible role-played by the simple catechins in coupled oxidations.

EC- Epicatechin, EGC-Epigallocatechin, ECQ-Epicatechin quinine,

TFI- Theaflavin intermediate, EGCQ- Epigallo catechin quinone.

During the manufacturing process, catechins are oxidized by catechol oxidase (polyphenol oxidase) in presence of oxygen (Fig 2.5) Such oxidized catechins then coupled to form theaflavin and thearubigens or are reduced by other compounds. Production of tea aroma is attributed to these oxidation processes.

#### 2.8.2 Flavor

Flavor involves taste (nonvolatile) and aroma (volatile compounds). Phenolic compounds, bitterness, caffeine and amino acids are the main contributors to taste and aroma of tea.

#### 2.8.3 Taste of tea

The characteristic taste of tea comprises a balanced mixture of astringency, bitterness,umami (brothy taste) and slight sweetness. Catechins and their derivatives are the principal contributors of astringency and bitterness. Caffeine has a simple bitterness. Aminoacids contributes to brothy taste with slight sweetness or bitterness, depending on aminoacids.

| Phenolic compounds                                      | astringency    | Bitterness      |
|---|----------------|-----------------|
| (-) –EC bitter and sweet after taste(45.5) <sup>a</sup> | Not astringent | 60 <sup>b</sup> |
| (-) -EGC bitter and sweet after taste(35.2)             | Not astringent | 35              |
| (-)-ECG bitter and astringent (35.2)                    | 50             | 20              |
| (-)-EGCG bitter and astringent (20.2)                   | 60             | 30              |
| (+) –C bitter and sweet after taste(51.0)               | Not astringent | 60              |
| Crude theaflavin (a natural mixture)                    | 60             | 75-100          |
| Theaflavin (pure)                                       | 80             | 75-100          |
| Theaflavin digallate                                    | 12.5           | Not determined  |
| Gallic acid   | Not astringent | Not bitter      |
| Tannic acid   | 20             | 80              |
| TF monogallate(a natural mixture)                       | 36             | 30-50           |

Table 2.3 Sensory evaluations of tea catechins and their derivatives

<sup>a</sup> Threshold level reported by Nakagawa (1970)

<sup>b</sup> Threshold level reported by Sanderson (1976)

#### 2.8.4 Aroma of tea

During the withering step, the processing of black tea, the glysidically bound aroma compounds are hydrolysed by the enzymes. In the rolling step, enzymatic reaction continues along with oxidation. End of the fermentation step considerable amount of alcoholic aroma compounds, methyl salicylate, and (E)-2-hexenal are found in fermented tea leaves.(Takeo, 1984; Yamanashi, et al, 1990). After rolling strecker degradation of aminoacids are also caused by oxidation.

#### 2.8.5 Tea quality parameters

Shade of colour in made tea and brightness in the infusion are two important attributes besides aroma and taste.

#### 2.8.5.1 Theaflavin(TF)

Theaflavins are golden yellow pigment, constitute around 0.5%- 2% of dry weight, depending on the type manufacture of black tea. TF are responsible for the development of bright colour and brisk taste of the liquor. These compounds are mostly formed during the maceration stages in black tea manufacture. These groups of compounds contribute about 30% of the total colour and are the result of rolling and fermentation. Theaflavins also provides briskness, freshness and aliveness tot the infusion, which is highly valued in taster's parlance. Teas having maximum TF and a balanced TR/TF ratio (10 to 12) with optimum level of briskness and color indices will definitely have a better quality.

#### 2.8.4.2 Thearubigens (TR)

Thearubigens (TR) are the orange brown compounds constituting about 6-18% of dry weight and formed during the manufacturing process of the tea. They are heterogenous complexes and are responsible taste, total colour and body of liquor. They contribute around 35% of the total colour and also plays a significant role in brown appearance of made tea.

#### 2.8.5 Evaluation of tea quality

-The quality of made tea is mainly depend on the presence of desirable compounds, undesirable compounds, feeling and sensitiveness of taste. Tea tasters evalute tea quality by different number of parameters and they use different terms to express the quality (Wickramasinghe, 1978). However in biochemical the ratio between the ratio between TR/TF is used to evaluate the quality of tea.

#### CHAPTER 03

#### **3.1 MATERIALS**

Tender tealeaves ECM unit (Environment control miniature manufacturing unit) Rollers Fermentation trays Dryers Spectrophotometer

#### **3.2 METHODOLOGY**

### 3.2.1 Location

The experiment was conducted at St. Coombs Estate, Talawakelle, managed by Tea Research Institute of Sri Lanka. The area comes under  $WU_3$  (Agro ecological zone).

Climatic conditions experienced during the period was,

| Average rainfall  | 18mm/month |
|-------------------|------------|
| Temperature       | 15-18 ° C  |
| Relative humidity | 80-85%     |

#### 3.2.2 Selection of cultivar

Cultivar DT1 (belong to high quality tea cultivar) and TRI 2025 (belongs to poor quality tea cultivar) were used for this experiment.

#### 3.2.3 Sampling

4 Kg of fine plucked tender leaves (normally except coarse leaves and damaged leaves) were selected for each treatment.

#### 3.2.4 Treatments

4.0 ° F - control 5.0 ° F 6.0 ° F 7.0 ° F 8.0 ° F

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#### 3.2.5 Time of plucking

Plucking time was allocated from 10.30 a.m to 12.30 p.m with a six day plucking round.

## 3.2.6 Procedure

Plucked green leaves were transported to the factory by the Lorry.

4 Kg of tealeaves were selected from the factory.

50 g of green leaf sample was put in to the oven for testing initial moisture content at 103 ° C for 6 hrs.

After the withering process the tealeaves were tested for moisture content. At last the dried made tea samples were selected for moisture content.

#### 3.2.7 Miniature manufacture

The selected green leaves (without coarse leaves and damaged leaves) were kept in two ECM for withering process for 16 hrs. One of the ECM was set for hygrometry difference 4 ° F. This hygrometry difference is considered as control. Other ECM unit was set for treatment hygrometry differences.

Samples were weighed separately before loaded it in to the miniature rollers. First dhool was extracted after 30 minutes of rolling process. And the big bulk was rolled again and second dhool was extracted after another 30 minutes.

Fermentation was scheduled for 2  $\frac{1}{2}$  hrs. During the fermentation process the wet bulb and dry bulb temperature maintained at 3 ° F. End point of fermentation level was decided by desirable aroma and colour change.

When the dhool was attained correct fermentation level, it was fired using miniature tea dryer. The inlet temperature was maintained at 190-200 ° F and exhaust temperature was maintained at 130-140 ° F during the 21 minutes of firing period. Then fired dhool was weighed separately and sealed air tight in polythene bags.

Then dhool was hand sieved (Sieve number 12 and 14) in a closed environment under 65 % relative humidity conditions to separate BOP tea grade. Then fibre and stalk were removed and 10 g of sample of control and treatment ,was put in to oven to determine final moisture content.

## 3.2.8 Chemical analysis

Laboratory analysis was conducted at Technology Division of TRI. Two samples from each category were tested for TF, TR, Total colour and brightness after six days from the day of manufacturing. Robert and Smith method (1962) was used for the analysis.

#### 3.2.9 Experimental design

Experiment was laid out as a Randomized completely blocked design (RCBD) with five treatments and four blocks.

#### 3.3.0 Data analysis

All collected data was statistically analyzed by using Statistical Analysis System (SAS). The LSD procedure was used to compare treatment means. Difference between treatment means was considered at significance level P<0.05.

## **CHAPTER 04**

## **RESULTS AND DISCUSSION**

## 4.1 Results

(1) Variation of TF (%) content with control in clone TRI 2025 and DT1

|           | TF %    |                  |         |                                   |  |
|-----------|---------|------------------|---------|-----------------------------------|--|
|           |         | DT1              |         | TRI 2025                          |  |
| H.D (° F) |         | Difference (° F) |         | Diff <b>er</b> en <b>ce</b> (° F) |  |
| 4         | 1.3821  |                  | 1.3043  |                                   |  |
| 5         | 1.45855 | 0.07645          | 1.326   | 0.0217                            |  |
| 4         | 1.0425  |                  | 0.83925 |                                   |  |
| 6         | 1.1045  | 0.062            | 1.0119  | 0.17265                           |  |
| 4         | 1.1505  |                  | 0.6834  |                                   |  |
| 7         | 0.7763  | -0.3742          | 0.6035  | -0.0799                           |  |
| 4         | 1.14075 |                  | 0.8539  |                                   |  |
| 8         | 0.8269  | -0.31385         | 0.60695 | -0.24695                          |  |

(2) Variation of TR (%) content with control in clone TRI 2025 and DT1

|           |        | TR %             |         |            |
|-----------|--------|------------------|---------|------------|
|           |        | DT1              | TRI     | 2025       |
|           |        |                  |         | Difference |
| H.D (° F) |        | Difference (° F) |         | (° F)      |
| 4         | 18.32  |                  | 15.0678 |            |
| 5         | 18.75  | 0.43             | 15.3137 | 0.2459     |
| 4         | 15.01  |                  | 12.5592 |            |
| 6         | 13.075 | -1.945           | 14.2447 | 1.6855     |
| 4         | 15.838 |                  | 11.6961 |            |
| 7         | 13.767 | -2.071           | 11.205  | -0.4911    |
| <b>4</b>  | 16.02  |                  | 16.6    |            |
| 8         | 12.245 | -3.775           | 15.04   | -1.56      |
|           |        |                  |         |            |

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# (3) Variation of Total colour (%) content with control in clone TRI 2025 and DT1

|           |         | TC %            |        |                  |  |
|-----------|---------|-----------------|--------|------------------|--|
|           |         | DT1             |        | TRI 2025         |  |
| H.D (° F) |         | Difference (° F | )      | Difference (° F) |  |
| 4         | 5.56245 |                 | 3.525  |                  |  |
| 5         | 6.4156  | 0.85315         | 3.698  | 0.173            |  |
| 4         | 8.514   |                 | 2.478  |                  |  |
| 6         | 3.2153  | -5.2987         | 3.365  | 0.887            |  |
| 4         | 4.6906  |                 | 2.9415 |                  |  |
| 7         | 3.0687  | -1.5219         | 2.8315 | 0.11             |  |
| 4         | 4.9608  |                 | 3.6325 |                  |  |
| 8         | 3.045   | -1.9158         | 2.7935 | -0.839           |  |
|           |         |                 |        |                  |  |

(4)Variation of brightness (%) content with control in clone TRI 2025 and DT1

|           |        | DT1              |        | TRI 2025         |
|-----------|--------|------------------|--------|------------------|
| H.D (° F) |        | Difference (° F) |        | Difference (° F) |
| 4         | 23.425 |                  | 29.395 |                  |
| 5         | 21.05  | -2.375           | 27.495 | -1.9             |
| 4         | 10.704 |                  | 27.57  |                  |
| 6         | 13.48  | 2.776            | 25.115 | -2.455           |
| 4         | 22.53  |                  | 21.095 |                  |
| 7         | 20.54  | -1.99            | 19.875 | -1.22            |
| 4         | 22.03  |                  | 18.72  |                  |
| 8         | 23.225 | 1.195            | 17.735 | -0.985           |
|           |        |                  |        |                  |

## Brightness %

## (5) Variation of TF: TR in clone TRI 2025 and DT1

| H.D (° F) | . TF    | =: TR    |
|-----------|---------|----------|
|           | DT1     | TRI 2025 |
| 4         | 01:13.3 | 01:11.5  |
| 5         | 01:12.9 | 01:11.5  |
| 4         | 01:14.4 | 01:14.6  |
| 6         | 01:11.8 | 01:14.1  |
| 4         | 01:13.8 | 01:17.1  |
| 7         | 01:17.7 | 01:18.6  |
| 4         | 01:14.0 | 01:19.4  |
| 8         | 01:14.8 | 01:24.8  |
|           |         |          |

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# (6) Variation of moisture content in different stages of manufacture (TRI 2025)

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| H.D (° F) | Moisture content% |               |          |  |
|-----------|-------------------|---------------|----------|--|
|           | Greenleaf         | withered leaf | Made tea |  |
| 4         | 75.95             | 59.67         | 4.15     |  |
| 5         | 75.95             | 56.45         | 4.3      |  |
| 4         | 79.17             | 57.2          | 3.85     |  |
| 6         | 79.17             | 55.31         | 4.7      |  |
| 4         | 77.34             | 64.58         | 5.65     |  |
| 7         | 77.34             | 58.56         | 4.45     |  |
| 4         | 76.93             | 60.64         | . 5.15   |  |
| 8         | 76.93             | 52.42         | 4.5      |  |

## (7) Variation of moisture content in different stages of manufacture (DT 1)

| H.D (° F) | Moisture content% |               |          |  |
|-----------|-------------------|---------------|----------|--|
|           | Greenleaf         | withered leaf | Made tea |  |
| 4         | 77.75             | 60.64         | 3.9      |  |
| 5         | 77.75             | 57.42         | 3.4      |  |
| 4         | 76.7              | 63.24         | 4.55     |  |
| 6         | 76.7              | 52.36         | 4.2      |  |
| 4         | 77.21             | 62.64         | 4.7      |  |
| 7         | 77.21             | 57.875        | 3.65     |  |

| 4 | 77.42 | 63.42          | 4.05 |
|---|-------|----------------|------|
| 8 | 77.42 | 58. <b>9</b> 4 | 3.95 |

#### 4.2 Effect of withering rate on tea quality

The value of quality parameters obtained for the made tea samples with different hygrometry differences are significant, when it was statistically analyzed. It was tested at significant level P=0.05.

#### 4.3 Variation of TF (%) content with control in clone TRI 2025 and DT1

TF contribute towards the briskness and brightness of tea liquor. DT 1 is a high quality cultivar when compare to TRI 2025. There is a significant difference within the treatments. Made tea samples obtained from 1 <sup>st</sup> and 2 <sup>nd</sup> treatments shows (Fig 4.1) high value of theaflavin (TF) content when compare to control treatment (4 ° F). DT 1cultivars are narrow, erect leaved plants. Therefore the withering rate is higher than TRI 2025 clonal teas. The graph shows when expose to 1 <sup>st</sup> and 2 <sup>nd</sup> treatments results desirable increase of TF content in made tea samples. Exposure to 3 <sup>rd</sup> and 4<sup>th</sup> treatments leads to reduction in TF content. Because it is due to further conversion of TF into TR. Therefore the content of TF is low when the hygrometry difference is high that means when the withering rate increases the TF content leads to reduce.

TRI 2025 is a poor quality clone. TRI 2025 also follows the same manner.1  $^{st}$  and 2<sup>nd</sup> treatments show high value of TF content than 3<sup>rd</sup> and 4<sup>th</sup> treatments. But the value of TF content is low when comparing to DT 1 clone. Because the content of polyphenol is low in TRI 2025 cultivar than DT 1 cultivar. This is due to good quality characters of DT 1 clone.

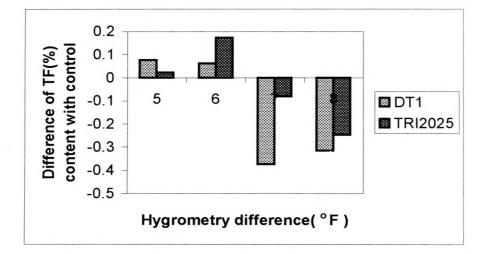


Fig 4.1 Variation of TF (%) content with control in clone TRI 2025 and DT1

1<sup>st</sup> treatment - 5° F, 2<sup>nd</sup> treatment - 6° F, 3<sup>rd</sup> treatment - 7° F, 4<sup>th</sup> treatment - 8° F

4.4 Variation of TR (%) content with control in clone TRI 2025 and DT1

For the DT1 clone shows significant difference of TR content for 1<sup>st</sup> treatment. There is high value of TR content for 1<sup>st</sup> treatment than other treatments. This result is due to high value of TF content because of the conversion of TF into TR.TRI 2025 shows (Fig 4.2) significant difference for 2<sup>nd</sup> treatment.

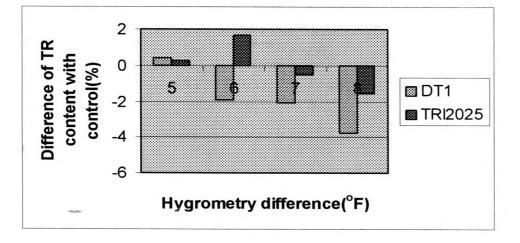


Fig 4.2 Variation of TR (%) content with control in clone TRI 2025 and DT1  $1^{st}$  treatment - 5° F, 2<sup>nd</sup> treatment - 6° F, 3<sup>rd</sup> treatment - 7° F, 4<sup>th</sup> treatment - 8° F

## 4.5 Variation of TF: TR in clone TRI 2025 and DT1

The mean values obtained for TF: TR shows significant variation (P=0.05) 2nd treatment of DT1 cultivars and 1<sup>st</sup> treatment of TRI 2025 cultivars. The ratio of TF: TR values shows (Fig 4.3) variation of 1:10 and 1:12 range for these significant treatments.

The ratio of TF: TR for ideal liquor ranges in between 1:10 and 1:12. The  $3^{rd}$  and  $4^{th}$  treatments show great deviation from  $1^{st}$  and  $2^{nd}$  treatments with a high value of TF: TR. This results poor liquor characteristics for tea with more brightness and less colour.

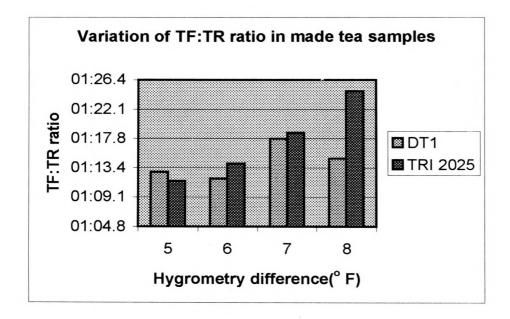


Fig 4.3 Variation of TF: TR in clone TRI 2025 and DT1

1<sup>st</sup> treatment - 5° F, 2<sup>nd</sup> treatment - 6° F, 3<sup>rd</sup> treatment - 7° F, 4<sup>th</sup> treatment - 8° F

4.5 Variation of Total colour (%) content with control in clone TRI 2025

and DT1

 $1^{st}$  treatment shows significant difference than other treatments for DT1 cultivars. TR is the main contributor for the liquor colour. For the TRI 2025  $1^{st}$  and  $2^{nd}$  treatments shows significant difference.

 $3^{rd}$  and  $4^{th}$  treatments show great deviations than  $1^{st}$  and  $2^{nd}$  treatments. But the overall graph shows (Fig 4.4) high total colour content in DT1 than TRI2025. This is due to good quality parameters of DT1 cultivar.

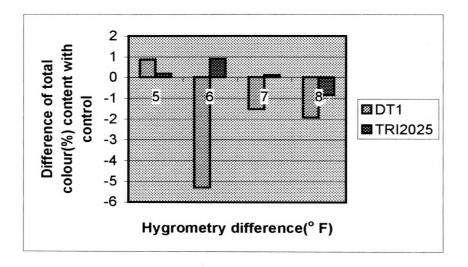


Fig 4.4 Variation of Total colour (%) content with control in clone TRI 2025 and DT1

1<sup>st</sup> treatment - 5° F, 2<sup>nd</sup> treatment - 6° F, 3<sup>rd</sup> treatment - 7° F, 4<sup>th</sup> treatment - 8° F

## 4.6 Variation of brightness (%) content with control in clone TRI 2025

## and DT1

TF is the main contributor for the brightness of liquor. The TRI 2025 shows (Fig 4.5) low variation within the treatments. But DT 1 shows high percentage of brightness of liquor for the  $2^{nd}$  treatment. This is due to high content of TF value.

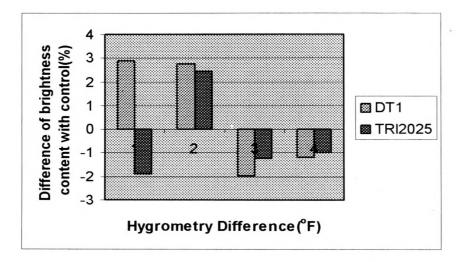


Fig 4.5 Variation of brightness (%) content with control in clone TRI 2025 and DT1

1<sup>st</sup> treatment - 5° F, 2<sup>nd</sup> treatment - 6° F, 3<sup>rd</sup> treatment - 7° F, 4<sup>th</sup> treatment - 8° F

4.8 Variation of moisture content in different stages of manufacture (TRI 2025)

This graphs shows (Fig 4.6, 4.7) a soft withering of tea leaves.soft withering moisture content ranges 58-65% moisture wetbasis.

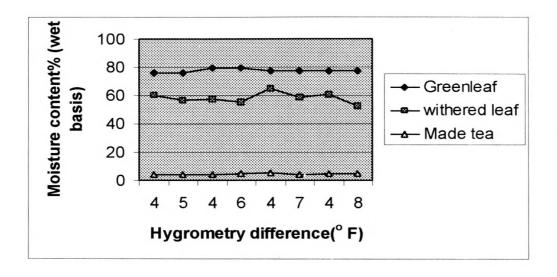
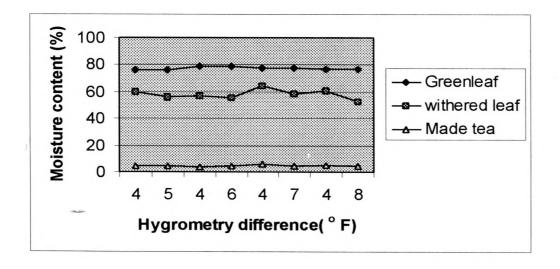
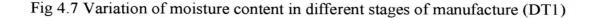


Fig 4.6 Variation of moisture content in different stages of manufacture (TRI 2025)





#### **CHAPTER 05**

## **CONCLUSIONS AND SUGGESTIONS**

#### **5.1 Conclusions**

According to this research study for DT1 clone the optimum hygrometry difference should be 5 ° F to achieve proper withering to get a good quality tea.

For TRI 2025 the optimum hygrometry difference should be 6 ° F to achieve proper withering and good quality tea.

Quality variation is more significant in withering rate of good quality cultivar DT 1 and poor quality cultivar TRI 2025.

## 5.2 Suggestions

This research should be carried out for other main tea grades such as BOPF and Dust No.1, and other manufacturing types such as Orthodox, rotorvane and CTC.

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#### Appendix 1

#### Method of Robert and Smith (1962)

The first method for analysis of Theaflavin and Thearubigin in tea liquor was based upon an Ethyl Acetate or Iso Butyl Methyl Ketone (IBMK) extraction.

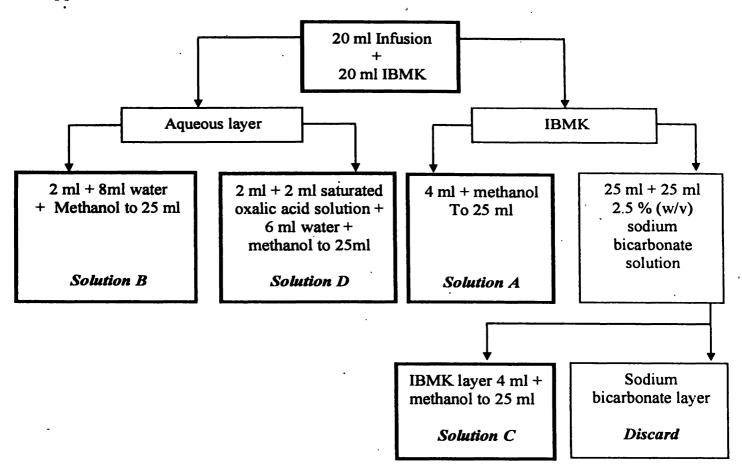
#### Procedure

Weigh 9 g of black tea leaf in to vacuum flask. Tare the flask and contents on a top pan balance and add 375 ml of boiling water from an overhead electric boiler. Stopper the flask and shake for 10 minutes in a mechanical shaker. Filter the infusion through cotton wool.

Extract 20 ml of infusion with 20 ml of IBMK and prepare the solution for spectrophotometer measurements as describe in flow diagram. Determine the extraction of solution A-D in spectrophotometer at 380 nm and 460 nm.

## Appendix 2

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Flow diagram for the analysis % of Theaflavin, Thearubigin, Total colour and Brightness in tea liquor.

41

Theaflavin (TF) % = (2.25) Ec

Thearubigin (TR) % =  $(1.77E_D + E_A - E_c)$  7.06

Total colour (TC) = 6.25 ( $E_{A'}$  + 2E  $_{B'}$ )

Brightness % = Ec'(100)

 $(E_{A'} + 2 E_{B'})$ 

EA EB Ec ED - Spectrophotometer readings at 380 nm

 $E_{A^{*}} \ E_{B^{*}} \ E_{C^{*}} \ E_{D^{*}}$  - Spectrophotometer readings at 460 nm

The SAS System 1 12:57 Monday, August 26, 2002

#### The GLM Procedure

Class Level Information

| Class     | Levels | Values      |
|-----------|--------|-------------|
| treatment | 4      | 1234        |
| qp        | 4      | TC TF TR br |

Number of observations 16

The SAS System 2 12:57 Monday, August 26, 2002

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The GLM Procedure
```

Dependent Variable: gain

| Source        |          | DF          | Sum of<br>Squares          | Mean Square F Val | Lue          |
|---------------|----------|-------------|----------------------------|-------------------|--------------|
| Model         |          | 6           | 32.29664335                | 5.38277389 1.     | .06          |
| Error         |          | 9           | 45.49745244                | 5.05527249        |              |
| Correct       | ed Total | 15          | 77.79409579                |                   |              |
|               |          | Source      | Pr                         | > F               | ·            |
|               |          | Model       | 0.4                        | 475               |              |
|               |          | Error       |                            |                   |              |
|               |          | Corrected T | otal                       |                   |              |
|               | R-Square | Coeff Var   | Root MSE                   | gain Mean         |              |
|               | 0.415155 | -423.9327   | 2.248393                   | -0.530366         |              |
| Source        |          | DF          | Type I SS                  | Mean Square F Val | Lue          |
| treatme<br>qp | ent      | 3<br>3      | 18.09114456<br>14.20549879 |                   | . 19<br>. 94 |

|                     | Source          | Pr                  | > F                          |
|---------------------|-----------------|---------------------|------------------------------|
|                     | treatment<br>qp |                     | 664<br>624                   |
| Source              | DF              | Type III SS         | Mean Square F Value          |
| treatment           | 3               | 18.09114456         | 6.03038152 1.19              |
|                     | Source          | Pr                  | > F                          |
|                     | treatment       | 0.3                 | 664                          |
|                     | The             | SAS System<br>12:57 | 3<br>Monday, August 26, 2002 |
|                     | The GL          | M Procedure         |                              |
| Dependent Variable: | gain            |                     |                              |
| Source              | DF              | Type III SS         | Mean Square F Value          |
| qp                  | 3               | 14.20549879         | 4.73516626 0.94              |
|                     | Source          | Pr                  | > F                          |
|                     | qp              | 0.4                 | 624                          |

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The SAS System 13:03 Monday, August 26, 2002

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## The GLM Procedure

## Class Level Information

| Class     | Levels | Values      |
|-----------|--------|-------------|
| treatment | 4      | 1234        |
| qp        | 4      | TC TF TR br |

## Number of observations 16

The SAS System 2 13:03 Monday, August 26, 2002

The GLM Procedure

Dependent Variable: gain

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| Source          | DF              | Sum of<br>Squares         | Mean Square F Value          |  |
|-----------------|-----------------|---------------------------|------------------------------|--|
| Model           | 6               | 11.70264448               | 1.95044075 2.36              |  |
| Error           | 9               | 7.44523618                | 0.82724846                   |  |
| Corrected Total | 15              | 19.14788066               |                              |  |
|                 | Source          | Pr                        | > F                          |  |
|                 | Model           | 0.1                       | 193                          |  |
|                 | Error           |                           |                              |  |
| 8-2-1           | Corrected Total |                           |                              |  |
| R-Square        | Coeff Var       | Root MSE                  | gain Mean                    |  |
| 0.611172        | -926.2037       | 0.909532                  | -0.098200                    |  |
| Source          | DF              | Type I SS                 | Mean Square F Value          |  |
| treatment<br>qp | 3<br>3          | 11.14094998<br>0.56169449 | 3.713649994.490.187231500.23 |  |

|                     | Source  |            | Pr > F           |         |  |  |  |
|---------------------|---|------------|------------------|---------|--|--|--|
|                     | treatment<br>qp                                   |            | 0.0345<br>0.8758 |         |  |  |  |
| Source              | DF  | Type III S | SS Mean Square   | F Value |  |  |  |
| treatment           | 3   | 11.1409499 | 3.71364999       | 4.49    |  |  |  |
|                     | Source Pr > F                                     |            |                  |         |  |  |  |
| -                   | treatment   | 0.0345     |                  |         |  |  |  |
|                     | The SAS System 3<br>13:03 Monday, August 26, 2002 |            |                  |         |  |  |  |
| The GLM Procedure   |   |            |                  |         |  |  |  |
| Dependent Variable: | gain  |            |                  |         |  |  |  |
| Source              | DF  | Type III S | SS Mean Square   | F Value |  |  |  |
| qp                  | 3   | 0.5616944  | 49 0.18723150    | 0.23    |  |  |  |
|                     | Source  | Pr > F     |                  |         |  |  |  |
|                     | qp  |            | 0.8758           |         |  |  |  |

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