

SEPARATION AND ANALYSIS OF COTTONSEED OIL DEODORIZER DISTILLATE (CODD) USING MOLECULAR DISTILLATION

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ABSTRACT

A molecular distillation (MD) method was developed to separate the unsaponifiables from Cottonseed oil deodorizer distillate (CODD), which contains free fatty acids (FFA), mono/diglycerides, tocopherols and sterols. Using a model KDL6 MD glass still with a 0.06 m² evaporator, five passes were conducted using different evaporator temperatures and with the condenser temperature held at 60 C and vacuum at 0.22 mmHg. With the feed rate of 0.2 kg/hr, at a temperature of 60 C, samples of the CODD were separated into the distillate and residue portion. Using a gas chromatography silylation method, quantification of the components was determined. The pass 4 distillate was composed of 90.8% of FFA and 4.3% of unsaponifiables and the residue had 63% unsaponifiables. Using this residue for stage-2, unsaponifiables were 84.5% of the distillate while the residue had 97.14% diglycerides. Molecular distillation is a solvent-free process, avoiding problems with toxicity. This laboratory method using a 2-stage process successfully distilled the unsaponifiables from the components of CODD and distilled the valuable tocopherols and sterols and the FFA.

INTRODUCTION

1) COTTONSEED OIL DEODORIZER DISTILLATE (CODD)

- is a by-product of edible cottonseed oil.
- is produced during refining, bleaching and deodorizing of cottonseed oil.
- contains a large amount of free fatty acids (FFA), some mono/diglycerides, and a collection of healthy compounds such as tocopherols and sterols ranging between 6-8% and 14-17%, respectively.

2) "SHORT PATH" MOLECULAR DISTILLATION (SPMD)

- is a technique for separating, purifying, and concentrating.
- easily separates heavy molecules at specific temperatures.
- decreases the heat exposure time of the volatile molecules.
- is advantages Fats and Oils, Foods/Flavors/ Fragrances, Chemicals/Petrochemicals/Polyphenols.

3) UNSAPONIFIABLES

- are fat and solvent-soluble components.
- includes health benefits:
 - Phytosterols:** anti-inflammatory, anti-cholesterol, anti-oxidative and anti-carcinogenic activities.
 - Tocopherols:** scavengers of lipid peroxy radicals, prevention of Alzheimer's disease and cancer.

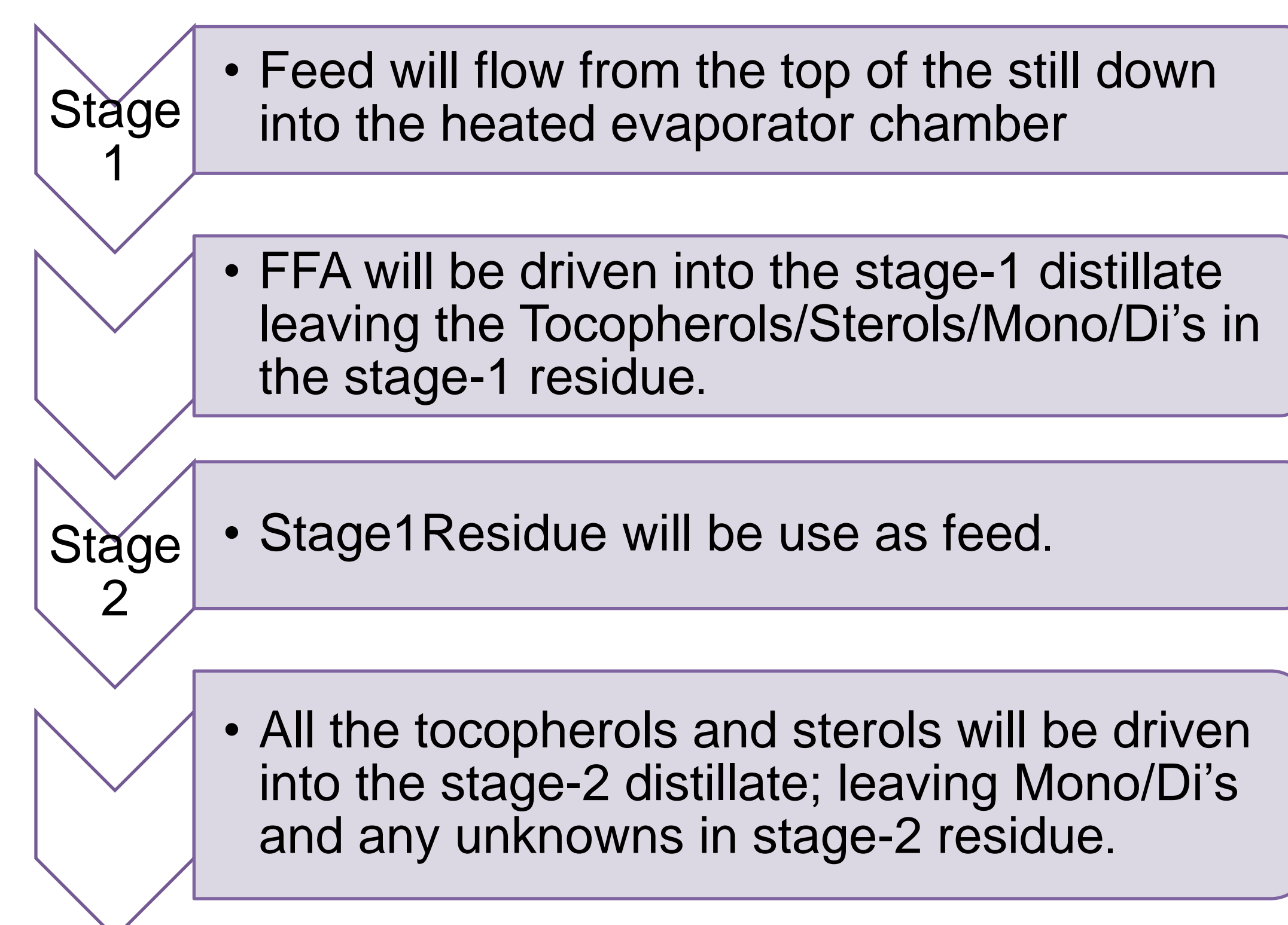
PURPOSE

The purpose of this study is:

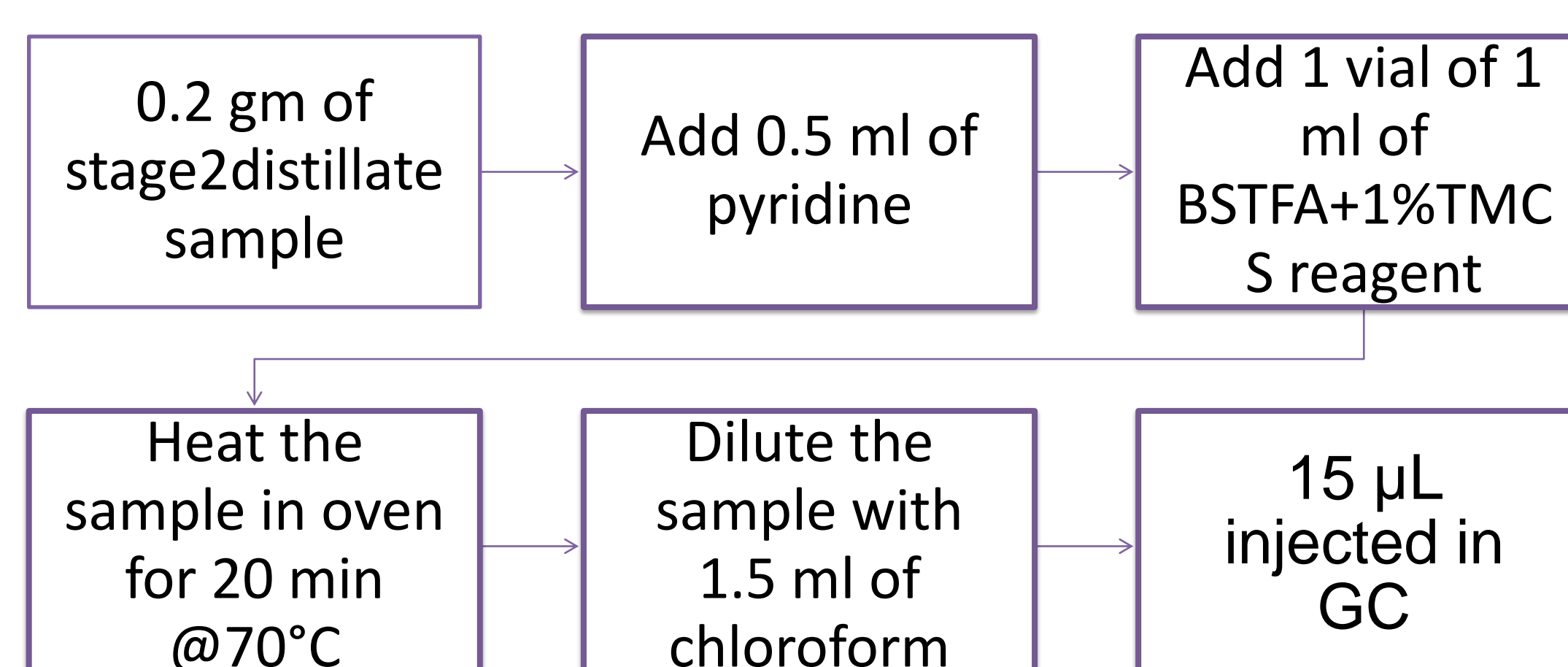
- To separate and recover the valuable unsaponifiables from cottonseed oil deodorized distillate specially the volatile compounds such as tocopherols and sterols.
- To achieve <1% FFA in stage-1 distillate and >95% purity of the tocopherols/sterols mixture in the stage-2 distillate.
- To validate the technique using the molecular distillation to separate and recover the unsaponifiables.

METHODOLOGY

1. Processing of CODD using SMPD:



2. Sample analysis using silylation:



3. Gas Chromatography:

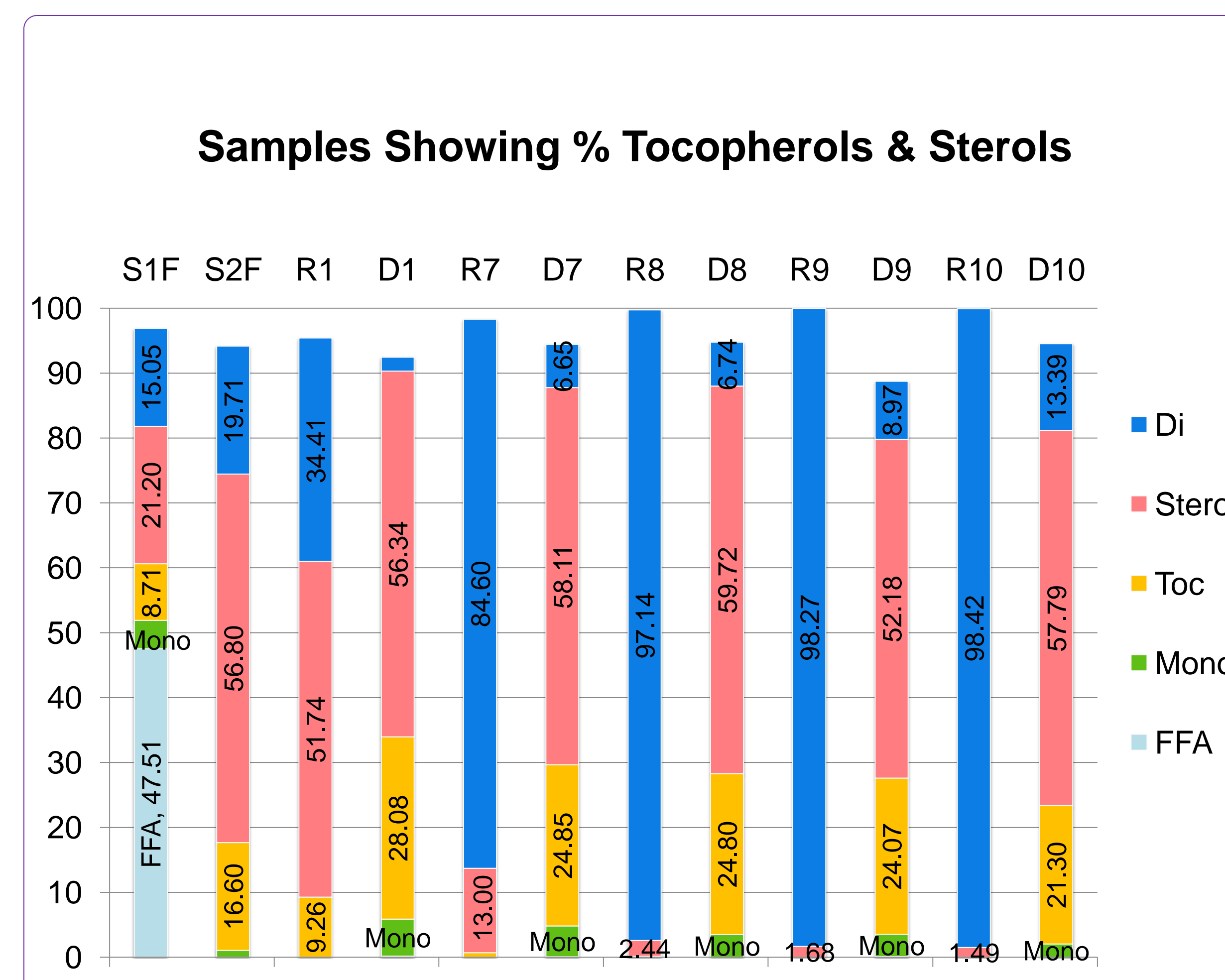
- Silylated samples will be injected on a capillary column CP-Sil 8 CB
- Detector: 0.1µm Flame Ionization Detector
- Temperature: injection temperature: 60 C, detector temperature: 360 C
- Carrier gas: nitrogen

RESULTS

Results of Molecular Distillation of CODD with a KDL6 glass still with 0.06 m²s Evaporator for stage-1

PARAMETERS:	1	2	3	4	5
Feed Rate (kg/hr)	0.2	0.2	0.2	0.2	0.2
Feed C	60	60	60	60	60
Evaporator C	135	145	155	165	175
RESULTS:					
% Yield distillate	26.4	43.8	52.2	55.6	59.2
% Yield residue	73.6	56.2	47.8	44.4	40.8
FFA content in the distillate (%)	83.1	83.4	83.1	80	73.3
FFA content in the residue (%)	32.3	16.2	4.6	2.2	1.4
Tocopherols content in the distillate (%)	0	0.3	1	2.1	3
Tocopherols content in the residue (%)	0	12.8	18.2	18.6	16.2
Sterol content in the distillate (%)	0	0.3	1.1	2.3	4.3
Sterol content in the residue (%)	0	26.2	34.8	44.4	51

Results of Molecular Distillation of CODD with a KDL6 glass still with 0.06 m²s Evaporator for stage-2



DISCUSSION

- In stage-1, five passes were used using five different evaporator temperatures.
- Various % yield of distillates and residues along with FFA, tocopherols and sterols were obtained which is given in the above table.
- The % FFA in the residue in stage-1 pass 4 & 5 were 2.2 and 1.4%, respectively. Almost all of the tocopherols and sterols volatilize into residue, leaving minor amount in distillate.
- % Yield distillate in stage-1 which is 55.6% and 59.2%, respectively in pass 4 and 5 respectively, shows complete removal of FFA.
- Stage-1 pass 4 had a good separation of unsaponifiables consisting of 18.6% of tocopherols and 44.4% of sterols.

DISCUSSION (contd.)

- Stage-1 pass 4 was used as a feed for 10 different passes.
- Stage-2 pass 8 had a good separation of unsaponifiables consisting of 24.80% tocopherols and 59.72% sterols, leaving all the 97.14% diglycerides with a negligible amount of tocopherols and 2% sterols in the residue.
- Stage-2 pass 8 was used as a feed at a temperature of 205 C and resulted in successful distillation of tocopherols and sterols.
- Stage-1 pass 4 and 5 produced acceptable results while stage-2 pass 8,9 and 10 showed a good recovery of tocopherols and sterols.
- Up to 45% of FFA were removed from CODD using wiped-film technique in 1st stage.
- The results indicate that a good recovery of unsaponifiables is obtainable.
- The FFA have a market value as a stand-alone product. The tocopherols and sterols mixture in the stage-2 distillate is also more concentrated and is now available as a stand-alone product.

CONCLUSION

- The stage-1 pass 4 distillate contained over 90.8% of FFA. The FFA were successfully separated.
- Stage-1 distillate gives a larger portion of FFA in the CODD which has a great market value.
- Stage-1 residue can be used as a feed for stage-2 to capture >95% of the tocopherols and sterols in the CODD which have a high market value of their own.
- 2-stage Molecular Distillation wiped-film technique is a successful process for purification and concentration of tocopherols and sterols, and removal of FFA.

ACKNOWLEDGEMENTS

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REFERENCES

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