REDESIGN AND ANALYSIS OF PRESAMPLER FOR GAS CHROMATOGRAPHER

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***Abstract*— The volatile is a substance which has boiling point up to 200°C and changes its state from solid or liquid to gas. The devices available for sampling are based on physical and chemical properties of molecules hence these are not complete. The gas chromatography is instrument used for volatile study but it requires only volatile matter to be entered into it. So the better sampling device is required to avoid nonvolatile matter. Thermal and kinetic desorption separate the large number of molecules and enhances the efficiency of instrument like GC-MS. A sampler using thermo-kinetic principle was manufactured. Study of sampler shows 25 percent increase in peaks when analysed with GC. The aim of current study is to improve the design of presampler and carry out the experimentation on each of the component of presampler. Also to reduce the time required for completing the one testing of volatile in GC.**

***Index Terms*— Condenser, Cooling, Desorpter, Heating, Heat transfer, peltier chips, volatile separation.**

I. INTRODUCTION

The report describes a new design approach that came from volatile separation. The conventional devices can separate volatiles of low boiling fraction. Volatile is a substance which can change state from a solid or liquid to vapour. Gas chromatography (GC) is the instrument used to study volatiles and it can analyse molecules whose boiling points are as high as 250°C or more. But it is necessary to make sure that Non-volatile material should not enter into the GC column. So there is a requirement of a safe sampling device for GC. Sampling device is separator unit, which will extract the molecules or volatiles from the given sample. The Prototype device for this is already manufactured, which is a POC (Proof of Concept). The goal of this report is to design the modular equipment and increase the heating and cooling rate of condenser and desorpter. For the purpose of heating purpose cartridge heaters are used with sensor for feedback control system. Also for cooling the purpose electric cooling chip are used, which works on peltier effect. The cooling of peltier chips hot side is done with the help of water cooler for faster cooling of the peltier.

For analysis of various system component the CFD software were used and for thermal analysis ANSYS is used.

II. LITERATURE REVIEW

Following literature describes the prototype of Volatile Extraction Device developed previously. This device works on thermo-Kinetic Principle of extraction. The equipment contains following parts:

1. PID Controller.
2. Electrical Circuit.
3. Flow and Temperature control.
4. Desorpter and Condenser.
5. Absorbent.
6. Heating and cooling Media.
7. Critical components of the system are design and geometry of Desorpter and Condenser, selection of heating and cooling media, absorbent material, and feedback control system for governing temperature, flow and Pressure.

A. *Description of Various Sub system:*

Desorpter:

Fig. 1 shows desorpter unit, it is a single piece unit carved out of 316 grade stainless steel (food and surgical grade, ideal for pressure reactors and boilers and is corrosion resistant) and fitted with screw cap for sample load. A GC septum or O rings secured airtight sealing. Holes are drilled in the body to accommodate the cartridge type heaters and temperature sensors. The heaters and sensors are connected to the thermostat and can be programmed from ambient temperature to 350°C in multi steps.

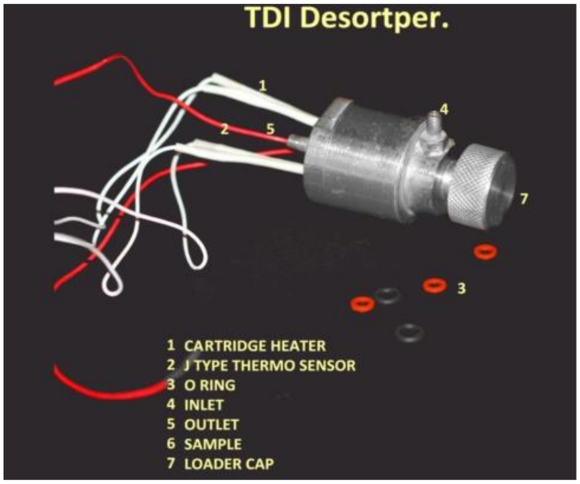


Fig. 1 old model of Desorpter

Condenser:

Fig.2 shows the condenser unit is slightly more complex than the desorpter both in functioning and in construction. The main body is 316 stainless steel cylinders with a 1cm x 6cm hole drilled in. The adsorbent material is packed in here. A special heater with inbuilt thermo sensors is wound around the condenser tightly. This heater is capable of heating the condenser to 250^{O}C ±1^{O}C in just 5 seconds. The cooling system consists of two aluminium boxes with cylindrical slit at its centre. These boxes are driven with a lever system to separate them from the condenser or to bring them close to the aluminium sleeve. The coolant used is a mixture of dry ice and acetone.

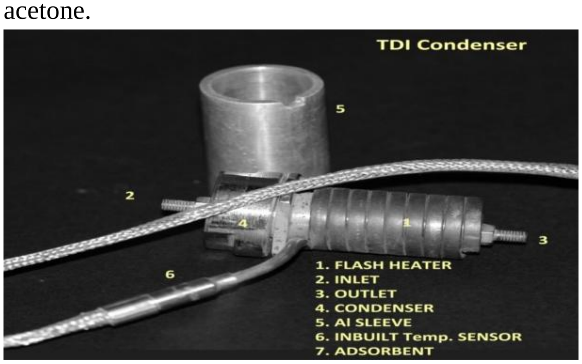


Fig. 2 old model of condenser

B. *Working*

A solid or semisolid sample containing volatiles is heated using multiple sources in a current of dry or moist (controlled) Nitrogen and the effluents are driven to a frigid cartridge pre-loaded with appropriate adsorbent. Once the adsorption is complete, the cartridge can be flash heated and the pneumatics drive the volatiles through appropriate membrane filters to the GC for analysis. Membrane filters are used to selectively detain water molecules and molecules of particular choice. Fig.1 shows the typical layout of existing system.

III. METHOD

Fig. 3 shows the presampler layout, in which the desorpter is heated using cartridge heater. The condenser is cooled faster and then condenser is flash heated to desired temperature. Various sensors are used to account for feedback control system such as pressure sensor, temperature sensor, and flow sensor. Also the solenoid valve is used to regulate the flow of nitrogen and volatile to the GC.

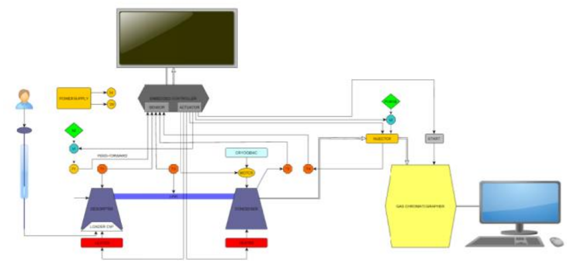
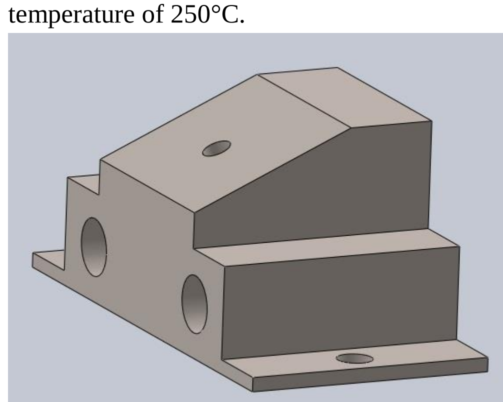


Fig. 3 Proposed presampler for GC.

A. *Desorpter Design*

Thermal analysis of old Desorpter:

Fig. 4 shows the CAD model of desorpter in which the sample is kept inside the cavity provided with threaded cap. At the inlet nitrogen gas of room temperature and pressure of 2 bar enters into the chamber and two Cartridge heaters of 150 watts is used. The material used is 316 grade stainless steel. These to heaters required time of 20 min to achieve temperature of 250°C.



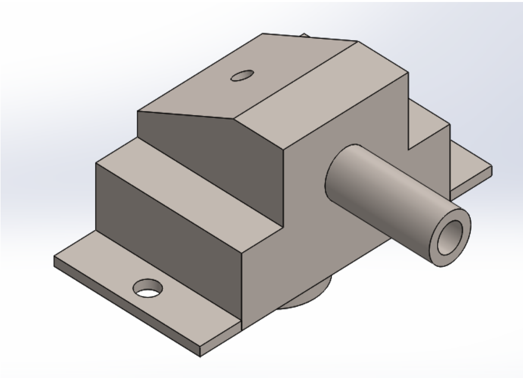


Fig. 4 CAD model of old desorpter.

Result:

The result shows that the time required for completing the heating process is nearly 20 min. also the gradually heating is required for the desorpter to remove the volatiles from mother matrix. The temperature profile fig. 5 shows that the uniform heating of desorpter is not achieved in minimum type.

Time taken by heaters to heat the desorpter: 10 min

It is observed that the experimental values and simulation values are matching. Hence the ANSYS analysis is valid.

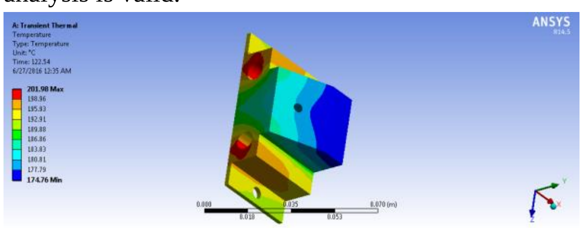


Fig. 5 thermal profile for old desorpter

New desorpter design:

The design of component is modified. Instead of two heaters slots we used four heater slots and also the weight of the desopter is reduced. The heating rate is increased so that the desorpter will be ready at room temperature for next sample loading. The new model is shown in fig. 6.

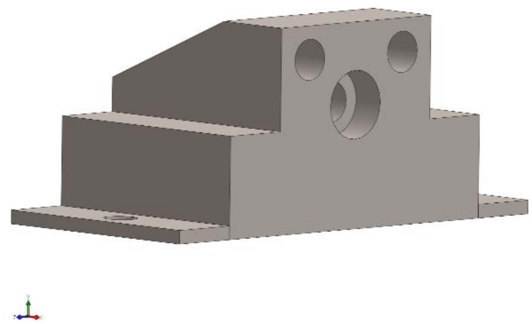


Fig. 6 modified Desorpter model

Heaters selection

To improve the heat generation rate and achieve gradual heating in desorpter also with minimum heating time we used following equation to raise temperature to 250°C in 3 min, the wattage of heater is given by:

$$KW = \frac{W_t.C_p.\Delta T}{3412.H} \qquad (1)$$

Wt = weight of material to be heated

Cp = Specific Heat of Aluminium alloy

T = Temperature rise

H = Heating time in hours.

It is decided to use four heaters of 125 watts power.

Thermal analysis of modified desorpter:

Since the time required for the heating is high and gradual heating is not obtaining the desorpter material is changed from 316 food grade stainless steel to aluminium 7075. Four cartridge heaters are used instead of two. The cartridge heater capacity is of 120 each. Thermal analysis shows in Fig. 7 the heating of desorpter up to 194°C is achieved in 107 sec. From the analysis it is cleared that with four heaters the uniform heating of desorpter is achieved which was not possible by the old desorpter design.

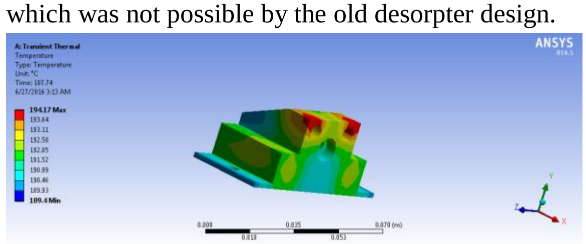


Fig.7 heating of new desorpter

B. *Condenser Design*

Thermal analysis of old condenser:

The function of condenser is to concentrate the volatiles by first cooling them up to −30°C and trapping it with in absorber. Absorber is a porous material which only allows nitrogen gas to pass through and will trap volatile. Again to release the volatiles absorber is flash heated up to 250°C. Then it delivers the volatile in GC machine. Fig 8 shows the 3D view of previous condenser.

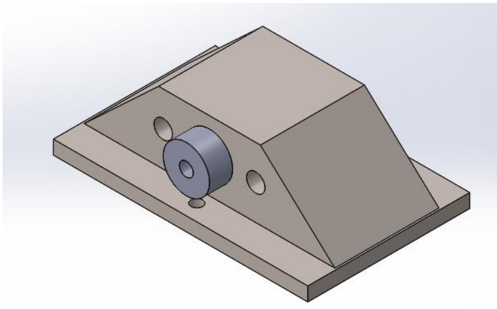


Fig. 8 3D model of condenser.

Old heat sink selection:

Application of heat sink in this is the same i.e. dissipation of heat. The junction to ambient resistance is calculated using following equation

$$R_{j-a} = \frac{T_j - T_a}{Q} \quad (2)$$

Rj-a = Resistance junction to ambient = 1.31 °C/W

Tj = Junction Temperature = 67 °C

Ta = Ambient temperature = 20 °C

Q = total heat developed = 36 W

Fig 9 Heat sink

The fin type of heat sink is used but the desired cooling is not achieved after experimentation. The peltier chips require higher cooling rate which is not achieved using air cooled heat sink. So to achieve cooling of hot side of peltier water cooler is used and the cooling up to -20 °C is achieved.

Thermal analysis of new heat dissipation system:

Heat generated by peltier chip

$$Q = m.C_p(T_2 - T_1) = 925 \text{ J} \quad (3)$$

Where, m = mass of device

Cp = specific heat of silicon

T2 = Hot side temperature of peltier chip

T1 = room temperature

Convective heat transfer coefficient:

$$G_r = \frac{gL^3\beta t_2 - t_1}{\vartheta^2} = 16.73\text{E}6 \quad (4)$$

Where,

g = acceleration due to gravity

ß = thermal expansion coefficient

v = kinematic viscosity

Prandtl number

$$P_r = \frac{\mu.C_p}{k} = 11.57 \quad (5)$$

Where,

μ = Dynamic viscosity of water

C_p= specific heat of water

k = thermal conductivity of water

Rayleigh number:

Ra = Gr.Pr = 19.35E7 (6)

Where,

Gr = Grashof number

Pr = Prandtl number

Nusselt number:

$$N_u = 0.27.R_a^{0.25} = 31.84 \quad (7)$$

Convective heat transfer coefficient:

$$h_c = \frac{N_u \times K}{L} = 41.53 \text{ W/m}^2\text{K} \quad (8)$$

Heating of condenser

After cooling the volatile and absorbed in condenser cavity then these volatiles are required to be release. For this purpose the flash heating is required ie 250°C in 2 min. the heaters wattage required is calculated below

$$W = \frac{W_t.C_p.\Delta T}{3412.H} = 245.36 \text{ W} \quad (9)$$

Wt = weight of material to be heated = 0.4Kg

Cp = specific heat of aluminium = 910 j/kgK

ΔT = temperature rise = 230°C

H = heating time in hours = 0.05

Heat transfer analysis for condenser:

The heating of old condenser is done by two cartridge heater of 250 watts. But it requires more than 5 min to reach 250°C. Thermal analysis is shown in Fig. 10. So the changes are made in following model fig. 11 where four cartridge heaters of 125 watts are used.

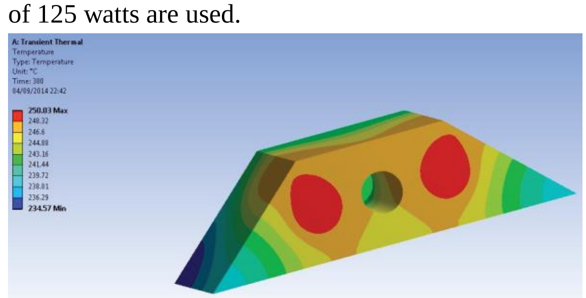


Fig. 10 temperature contour of old condenser

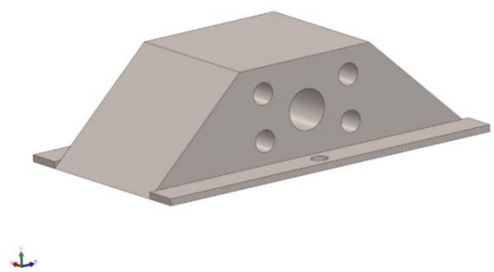


Fig. 11 3D model of New Condenser

The boundary condition applied is as following

Initial Temprature - 20°C

Maximum Temperature - 250°C

Heat transferred from heaters to condenser

$Q = m\,Cp(t2 – t1) = 83.45$ KW (10)

Where,

m = mass of condenser body

Cp = specific heat of aluminium

Result:

Fig. 12 shows that the temperature rise of 334 °C is achieved in just 300 sec. near cavity of condenser where the volatiles is accumulated and then by flash heating these volatiles will be release to GC. Uniform heating is achieved by four cartridge heater.

The analysis value and experimental values are matching hence the analysis is valid.

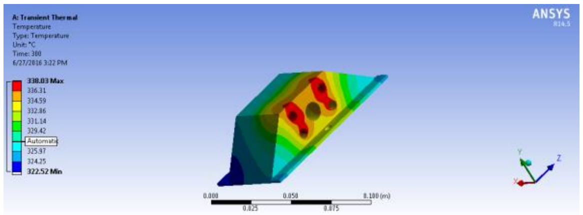


Fig. 12 Thermal analysis of new condenser.

IV. CONCLUSION

After analysis of desorpter, less time required for heating with four cartridge heater. Due to four heaters uniform heating is achieved. Also gradual heating of the desorpter is achieved with constant temperature gradient.

After analysis of condenser, flash heating of condenser is much more sophisticated when we used four heater and change the material to aluminium 7075 from 316 food grade stainless steel. Time required for flash heating is reduced to 3 min using new model of condenser. Also uniform heating along the cavity of condenser is achieved.

For cooling purpose of peltier chips hot side new cooling mechanism is introduced i.e. water cooler. Due to water cooling the temperature of condenser reduced to -10°C. To achieve this temperature cascading of peltier chips is done.

With this device the peaks of gas chromatography is increased by 25 %.

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