Spectroscopic Analysis of Expired and Pure Melmet

R.Kayalvizhi[a*], M.Sri Vidhya Rubini[a], A.Christy Ferdinand[a], G.Meenakshi[b]

[a]Department of Physics, St.Joseph's College of Arts & Science, Cuddalore.
[b]Department of Physics, K.M.C.P.G.S, Pondicherry.

[*]Corresponding **A**uthor: ramvizhi@yahoo.co.in

ABSTRACT

Spectroscopy is a perfect analyzer to find the elements of all matter. The application of spectroscopy has been used to interpret the effect of antibiotics and other medicines after its life duration. This has been studied by using the diabetics tablet melmet, expired for an year, to visualize the changes physically and chemically [1-3]. It has been observed that the occurrence of changes in color and reduction of smell and also the formation of new peaks and shift by XRD and UV, FTIR characterization respectively [4].

Keywords: *UV – Ultraviolet–Visible Spectroscopy   , FTIR - Fourier Transform Infrared Spectroscopy, XRD – X-ray diffraction, DSC - Differential Scanning Calorimetry*

1. INTRODUCTION

In this new era medicine plays a vital role to human beings else for all living beings by healing diseases. In that Melmet (metformin hydrochloride) plays a vital role in treatment of diabetics and poly cystic ovary diseases. Melmet contain metformin which is used for glucose tolerance and the chances increase of fertility. It helps to control blood sugar level. PCOD (Polycystic Ovary Disease) occur due to imbalance or abnormality in the hormones which can be control by Melmet [5].

2. EXPERIMENTAL

Many people especially who are living in remote areas consuming the medicine even after the expiry date due to the lack of knowledge in the medical field. Hence, it is essential to educate the

village people about the effect of consuming medicine after the expiry date. It is desired to take up some medicine after the expiry date to investigate their changes in physical and chemical properties [6]. One of such medicine was Melmet, it is used for the treatment of diabetics. Since, most of the human beings are suffering by this disease, that Melmet drug can be harmful to health in several ways after expiry [7, 8]. For this Melmet was made to cross its life duration for one year. This has been done by keeping it in an undisturbed place at normal room temperature. After that the pure Melmet and expired Melmet has been analyzed by Spectroscopic technique such as XRD, UV, FTIR, and thermal analysis DSC.

2.1. Characterization

The X-ray Diffractometer analysis has been carried out for the rapid identification and quantification of material by using Diffractometer system of XPERT-PRO at 2 theta position of 10.088 to 79.9381 2 with CuKα.
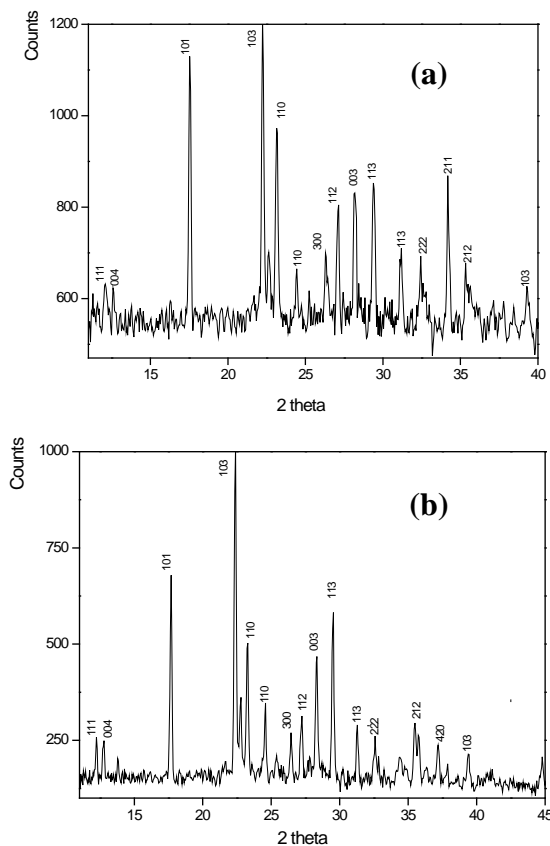

Fig.1 (a) and (b) shows the XRD analysis peak for pure
and expired Melmet sample

The optical absorption and transmission spectra have been recorded by using UV-VIS_NIR spectrometer in the range from 190-370nm as shown in Figure 1(a) and 1(b).
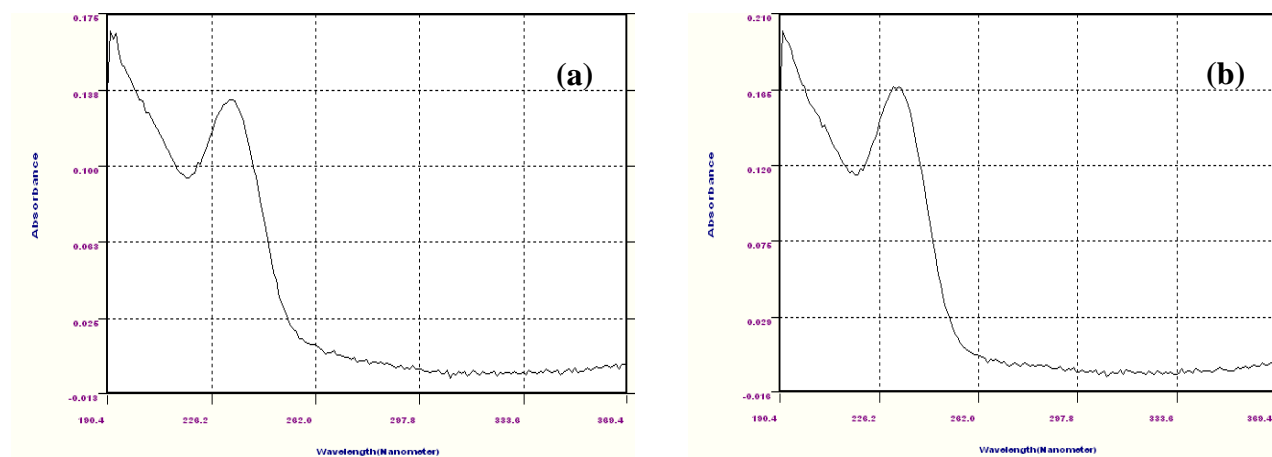
Fig.2 (a) and (b) shows the UV analysis peak for unexpired and expired Melmet sample

A non-destructive technique of FT-IR spectrometer has been made for pure and expired sample for around 3gm of drug mixed with KBr powder in the range of 4000-400cm^{-1}. The FT-IR spectrum for absorption and transmission has been shown in Table 1, Figure 2(a), (b) and 3(a), (b).

Table 1. Functional group and IR absorption bands.

| Functional group	IR absorption bands (cm^{-1})	
	Pure	Impure
N-H Asymmetric stretching	3369.7	3371.7
C-H Asymmetric stretching	3295.0	3293.1
N-H Symmetric stretching	3173.6	3178.0
Strongly bonded Hydrogen vibrations	2691.5	2691.0
C=C stretch	-	2182.2
N-H Torsion	1629.5	1634.3
C=C Asymmetric ring stretching	1578.5	1575.0
C-N Asymmetric stretching	1480.9	1482.1
C-N Symmetric stretching	1419.8	1418.0
N-H in –plane bending	1165.2	1165.4
N-H Twist	1062.5	1061.8
C-H out of plane bending	935.2	935.6
N-H Wagging	798.9	798.8
C-H Bend	735.0	734.4
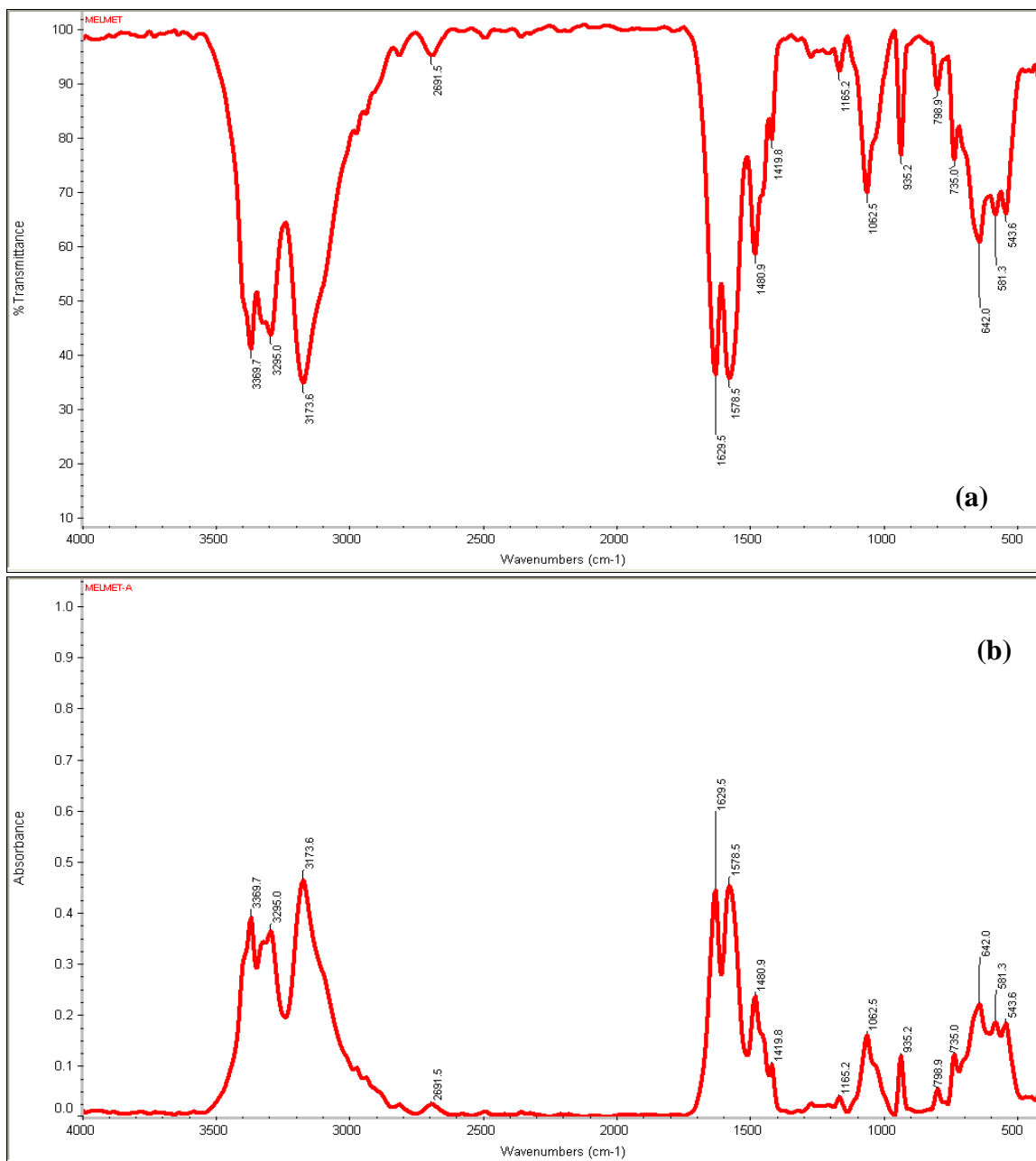
Fig 3 (a) & (b) shows the transmittance and absorbance peak for pure sample
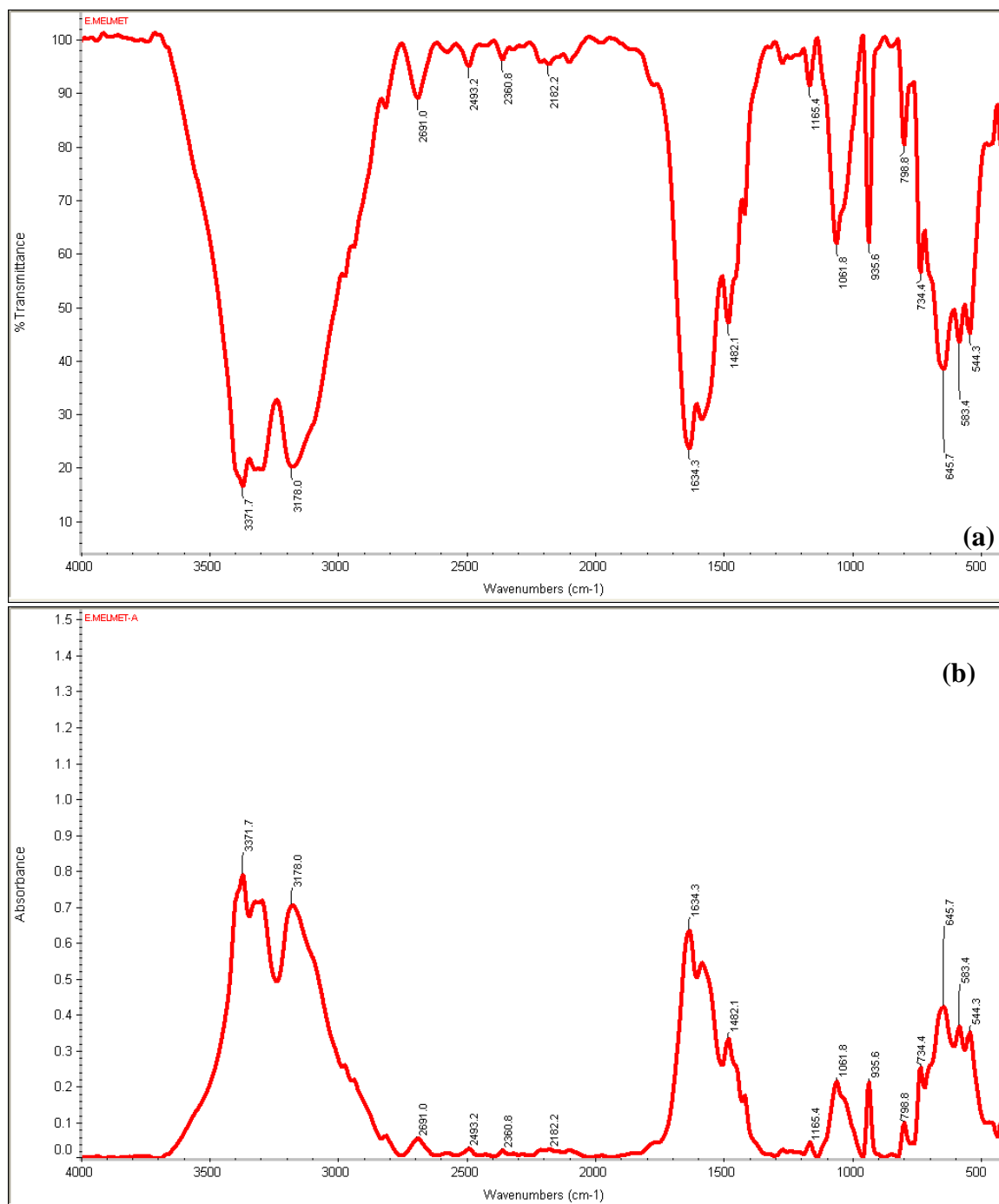
Fig 4 (a) & (b) shows the transmittance and absorbance peak for expired sample

Differential Scanning Calorimetry has been used to measure the amount of heat energy absorbed (endothermic) or released (exothermic) by the drug when it is heated or cooled processes.
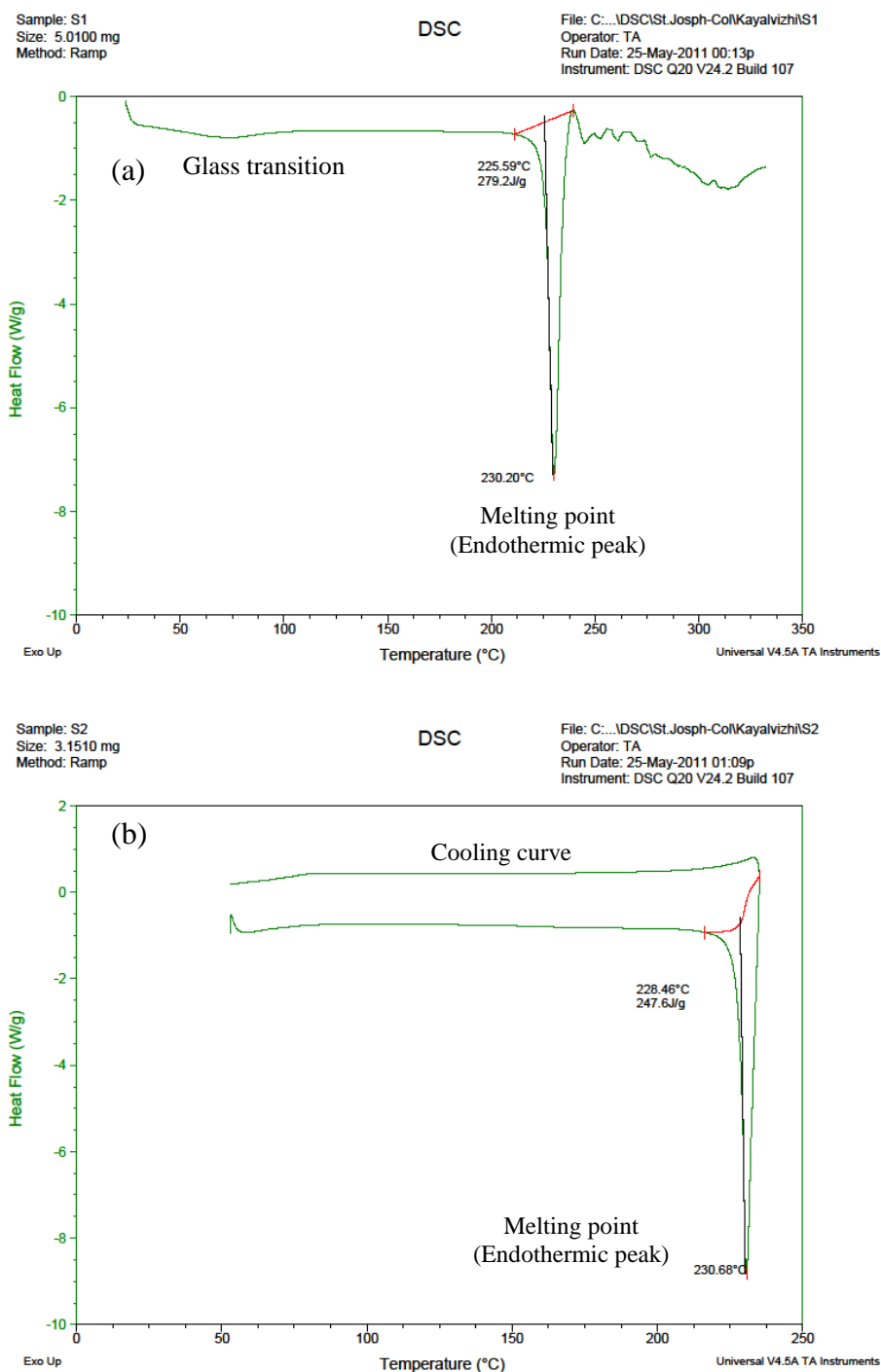

Fig 5 (a) & (b) DSC endothermic curve for pure and expired sample

3. RESULT

Expired drugs can be harmful to health in several ways; they can be unpredictable in effectiveness, simply ineffective, or even toxic.

According to the pharmaceutical industry, there are certain medications, such as nitroglycerine (used by heart patients for chest pain), insulin (used by diabetics to control blood sugar), and liquid medications such as antibiotics that degrade more quickly than other medications. This means that some of the effectiveness of the medication is lost, if it is used after the expiry date. In some cases risk with heart disease, diabetes, or serious infection. And while much of the original potency remains in other medications if used beyond their expiry date, their effectiveness depends in large part upon how the medication is stored. Factors shorten the lifespan of drug are moisture, increased temperature, manufacturing impurities, and, for some drugs, light and so on. Here the expired sample and unexpired sample were characterized by using XRD and UV to clarify why they are harmful after crossing the expiry date.

The excitation of X-ray in the life time crossed melmet (Fig 1a & 1b) has been found that in an expired sample some elements were newly raised. While comparing unexpired sample with expired drug the 2 theta values were increased slightly and correspondingly the d-spacing range also decreased in fraction. This is due to the incorporation of some other material in the original sample. The formation of new 211 planes has been seen in expired drug. The pyramidal plane 113 where dominated 112 pyramidal plane and 003 basal more in expired drug. i.e 112 and 003 planes have been compressed and 110 planes too. The decrease d-spacing values resulting that the drug molecule getting compress. And so it is not easy to get it diluted soon. Because of this condition it is contaminate in the kidney and developing a serious risk such as heart disease, diabetes, or serious infection.

From UV absorption pattern (Fig 2a & 2b) it is found that in an unexpired sample the two absorption peaks have been seen. First peak is around at 191nm with an absorbance range of 0.166 and the second peak is around 235nm with an absorbance range of 0.133. Whereas in an expired sample the absorbance range is slightly shifted above to 0.200 and 0.166 absorbance range respectively.

The complexity of infrared spectra in 1450 to 600 cm^{-1} (Fig 4a & 4b) region makes it difficult to assign all the absorption bands, and because of the unique patterns found there, it is often called the fingerprint region. Absorption bands in the 4000 to 1450 cm^{-1} region are usually due to stretching vibrations of diatomic units, and this is sometimes called the group frequency region.

A symmetric is a fashion at a wave number of 3173.6 cm^{-1}, 3178.0cm^{-1}, 1419.8cm^{-1}, 1418.0cm^{-1}, in which both bonds lengthen and contract together (in-phase), and the other in an asymmetric

fashion, in which one bond shortens while the other lengthens. The asymmetric stretch at a wave number of 3369.7cm^{-1}, 3371.7cm^{-1}, 3295.0 cm^{-1}, 3293.1 cm^{-1}, 1480.9 cm^{-1}, 1482.1 cm^{-1} is infrared active because there is a change in the molecular dipole moment during this vibration. (To be "active" means that absorption of a photon to excite the vibration is allowed by the rules of quantum mechanics.) [9].

Bond stretching 2182.2 cm^{-1} and bond bending 735.0 cm^{-1}, 734.4 cm^{-1}, more complicated molecules vibrate in rocking and twisting modes, which arise from combinations of bond bending in adjacent portions of a molecule.

Torsions at 1629.5 cm^{-1}, 1634.3 cm^{-1} involve changes in dihedral angles. This type of mode is analogous to twisting the lid off the top of a jar. No bonds are stretched, and no bond angles change, but the spatial relationship between the atoms attached to each of two adjacent atoms will change. [10] Stretching frequencies are higher than corresponding bending frequencies. (It is easier to bend a bond than to stretch or compress it.)At a wavenumber of 2691.5 cm^{-1}, 2691.0 cm^{-1}, a strong bond to hydrogen have higher stretching frequencies than those to heavier atoms. Triple bonds have higher stretching frequencies than corresponding double bonds, which in turn have higher frequencies than single bonds.

In a rocking, wagging or twisting coordinate the bond lengths within the groups involved do not change which is at 798.9 cm^{-1}, 798.8 cm^{-1}. The angles do. Rocking is distinguished from wagging by the fact that the atoms in the group stay in the same plane. [11]

Fig (5a & 5b) shows that the thermal analysis of pure and expired medicine. Pure melmet has been started to characterize from 10°C to 500°C for an amount of 5mg in Ramp method. The endothermic peak has been start at 225.59°C after the Glass Transition and exists its melting point at 230.20°C. After endothermic peak its starts to dispose in the form of brown colour. The same endothermic peak has been found in expired melmet but at high heat flow level. Cooling curve were tried to find its exothermic process but there is no result since it's not a crystalline material.

4. CONCLUSION

The experimental data indicates that the medicine which crossed its life time develops some new element which causes the changes in its physical and chemical condition. Using Xrd it's clearly found that the incorporation of elements in the form of new peak and shift in absorption peak explains its molecular contraction after expiry. And the small changes in its Stretching condition using FTIR and the shift in Heat Flow during Endothermic process explain the changes in its molecular bonding strength.

REFERENCES

[1] Hahnemann S., 1833, "The Organon of the Healing Art" Ed 5, aphorisms 5 and 217, ISBN 0879832282.

[2] Lord JM, Flight IHK, Norman RJ. "Metformin in polycystic ovary syndrome: systematic review and meta-analysis." BMJ. 2003;327(7421):951–3. doi:10.1136/bmj.327.7421.951. PMID 14576245. PMC 259161.

[3] Mishra. TK., 2005, Journal, Indian Academy of Clinical Medicine Volume 6, No. 4 .

[4] Lee J, Thompson E., 2007, "X-ray drug picture", The Homeopath (Northampton: The Society of Homeopaths) 26 (2): 43–48, ISSN 0263-3256.

[5] Ching-Ling Cheng, 2004, European Journal of Pharmaceutical Sciences, Volume 22, Issue 4, pp297-304

[6] Paul Nicklin, 1996, International Journal of Pharmaceutics, Volume 128, Issues 1-2, 29, pp 155-162

[7] Armagan Onal, 2009, European Journal of Medicinal Chemistry, Volume 44, Issue 12, pp 4998-5005

[8] I.I. Hamdan, 2010, Journal of Pharmaceutical and Biomedical Analysis, Volume 53, Issue 5, 15, Pages 1254-1257

[9] Schwartz A. T., et al., 1994, Chemistry in context DC, American chemical society, Washington

[10] Bratman, MD, Steven (1997). The Alternative Medicine Sourcebook. Lowell House. p. 7. ISBN 978-1-56565-626-0.

[11] G. Derosa, 2011, Diabetes Research and Clinical Practice, Volume 91, Issue 3, Pages 265-270.