Research Square

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A Thermogravimetric and Calorimetric Investigation of Natural and Artificial Sands

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Abstract

This thorough research looks at how four different sands behave when heated, those being the Beach Sand (B-01), Manufactured Sand (M-01), Red Sand (R-01) and River Sand (RS-01). It uses both Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) methods to figure out their breakdown due to heat, energy signatures and what phases they transform during heating up to the temperature of 1200°C. It was found that there are big differences in the way these sands react from minerals, how pure they are and what type of earth they come from. River sand had high exothermic reaction at about 594.5°C and around 597.3°C, resulting in large energy outflows like 1022.8 J/g then 267.9 J/g, mainly because of the α-β quartz change. Manufactured sand kept its stability under heating up to 750°C but soon after, strong exothermic activity happened at roughly an 945°C. Minor break down was present inside Beach sand at 522.3°C. For the red sand, it activated thermally at wide temperature intervals. These such results, it adds an important information for picking materials for building purposes, foundry work refractory making and any heat-related industries needing reliable thermal performance.

1. INTRODUCTION

1.1 Background and Importance

Sand is one of the most extensively consumed natural materials worldwide, with an estimated annual usage exceeding 50 billion metric tons across construction, industrial, and technical applications [1]. Because there is a rising shortage of natural river sand, as well as worries about environmental issues tied to its mining, there is an bigger interest in using other types of sand, like the manufactured sand, sands from beaches and special sands for example red sand[2] .All these sand types have unique minerals and chemistry which makes a change to the way they act to heat, which is important for uses at high temperatures.[3]

Around 95% of sand in the world is used by the construction field, especially for producing things like mortar, asphalt and concrete[1].But now, new uses like foundry sand molds, refractory walls, geothermal technologies and fireproof products, need more detailed information about how sand behaves in heat. Although much research is done on the physical or mechanical behavior of the sand, not a lot of direct comparison studies exist for thermal properties with modern analytic tools.[5] This missing information is important as new kinds of sand get used and their reactions for heat may be different.

1.2 Earlier Studies and Gaps in Knowledge

Older research that studied the thermal features of sands mainly put attention to just a single application or kind of sand[6]. Some papers about foundry sand discussed thermal expansion, but in construction applications the research focused more mainly on how sand reacts with an alkali-silica and how long it withstands. Older studies which examined heat-related aspects of the sands usually only looked at one sand type or for a purpose[6]. A few articles regarding foundry sands have talked about the thermal

expansion properties, but with sands used for construction, most investigations put most focus on the relationship with the alkali-silica as well as how durable it stays with time.. Manufactured sand, which is selling more in the market, has not gotten as much recognition from the researchers for its thermal performance. What is more, the published articles are hardly doing direct comparisons between natural and artificial sand samples while using TGA-DSC tests together over the entire important temperature range for industry (such as up to 1200 degree Celsius) [4], [5].Some important works were written, like by Smyth (2004) who discussed quartz transformation, Mackenzie (1970) who worked mainly on basics of thermal test methods and there are also studies by Chowdhury et al. (2015) who looked at how particle size matters. However, they all check different details instead of giving the big picture of heat behaviors for more sands. In response, this current study tries to fill the missing pieces by systematic comparative tests of the four sand types that are used commercially.

1.3 Research Objectives

The aim for this study focuses to:

1. Describe and make a comparison of thermal decomposition behavior for beach sand, manufactured sand, red sand, river sand by using the TGA/DTG investigation.
2. Find and measure the endothermic as well as exothermic actions with DSC between 25 up to an 1200 degrees Celsius [5].
3. Connect thermal situations to their mineral compositions and impurity present.
4. Check thermal stability levels for each of sands.
5. Look at industrial uses where the resisting heat is needed.[4]

1.4 Scope and Structure

This study lays out experimental methods, the specific outcomes, and a big discussion about the heat responses. Section 2 explains things about the materials and how experiments were conducted. Section 3 gives a TGA/DTG data, but Section 4 is about results found by the DSC. Section 5 brings a mixed review that links thermal events to material specifics.Section 6 looks at how the findings could be used and Section 7 ends with the conclusions and suggestions. At last, there is the acknowledgment with references.

2. MATERIALS AND METHADOLOGY

2.1 Material Collection and Preparation

This research used four different sand kinds picked as main groups for industrial use.

2.1.1 Beach Sand (B-01):

This sand got picked up from coast at Chennai Marina. It has rounded shapes, with salt amounts and some organic dirtiness. The sample was cleaned a lot with deionized water for getting rid of salts that can be solved in the water. The mineral composition inside the sand was kept.

2.1.2 Manufactured Sand (M-01):

Created by smashing a granite rock using machines from Central part TamilNadu Coimbatore. It is an example of industrial material that has pieces with sharp edges and size of grains was controlled[9]

2.1.3 Red Sand (R-01):

Came from Southern Tamil Nadu and different because it has so much iron oxide that makes it red. It also usually has both clay minerals and feldspars that are already weathered.

2.1.4 River Sand (RS-01):

Gotten from a Cauvery river. It has grains from sub-rounded up to rounded shapes, with many types of minerals like quartz, feldspars and some extra minerals. These sands were all dried at 105°C for one day to get out water,made uniform by dividing and mixing, and put through sieves to keep grain size from 300 to 600 μm for thermal test regularity.[12]

2.2 Mineralogical and Chemical Characterization

Samples had some primary characterizations before the thermal examination was carried out:

- An X-ray Fluorescence (XRF) checked elements in the material.
- X-ray Diffraction (XRD) was used for knowing what crystals were present.
- Loss on Ignition (LOI) experiments were done at 1000°C.
- Scanning Electron Microscopy (SEM) helped observing the morphology.

These different tests gave important background for understanding how thermal reactions occurred especially when talking about impurity levels and the mineral types.

2.3 Thermal Analysis Techniques

2.3.1 Simultaneous TGA-DSC Device:

Tests were carried out using simultaneous thermal analyzer, [Instrument Model, like Netzsch STA 449 F3 Jupiter] [14], which comes with:

- The microbalance has sensitivity of 0.1 microgram
- Temperature can be set from normal room temperature up to maximum 1600°C
- Heating speeds can be changed in range 1 to 50 K per minute
- Possible atmosphere settings: Inert (for example N_2 gas), oxidizing (artificial air type) and can be reducing as well

The DSC detector responds to lower than 1 micro Watt.

2.3.2 Experimental Setup :

The following steps are given below

1. Each sample used in the machine weighed about an 25 ± 0.5 mg
2. Alumina (chemical symbol Al_2O_3) crucible with a lid used to contain the material
3. Standard test done with temperature rising by 10°C per minute for comparing
4. Temperature changes between 25°C to 1200°C is made
5. Atmosphere handled by high-purity nitrogen flow, 50 mL per minute
6. Reference point is just empty crucible made of alumina
7. Device is calibrated with some certified reference things (Indium, Tin, Aluminum and Zinc) for temperature and sensitivity tracking

2.3.3 Data Collection and Post-Processing:

Data were collected in frequency 1 Hz. Analysis of results did steps like:

- Baseline adjustment is performed using blank crucible data
- Smoothening by Savitzky-Golay five-point window method applied to curves [13]
- Identification and area calculation of peaks are done with non-public software
- DTG graphs were made using derivative formulas

To confirm, every sample was measured three times

2.3.4 Other Analytical Techniques

To help explain the thermal experiment data:

- The FTIR method finds what functional groups are present
- Samples after heating are checked by XRD for changes of phase
- EDS used to make elemental maps on the heat-treated sample remains

2.4 Extra Methods for Analysis

Fourier Transform Infrared Spectroscopy (FTIR) has function to figure out functional groups which are inside samples.Heat-treated samples get tested with an X-ray diffraction (XRD) to notice any phase transitions. Also, the Energy Dispersive Spectroscopy (EDS) checks heat-treated residues so elemental spread can be analyzed.[15]

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3. THERMOGRAVIMETRIC ANALYSIS RESULTS

3.1 Thermal Behaviour of Beach Sand (B- 01)

G-DTG-DSC curves measured for a sample B-01 (see Fig. 3.1) show that there are different steps of losing mass, which together add up by a factor of 65 to 70 percent by weight. This suggests sand from the beaches are not uniform, mainly a quartz (SiO_2) with some clay additions like kaolinite and carbonates phases like calcite originating from biogenic sources [16]. When heated at 1200°C, what remains is more or less 33 percent of mass, which fits strong silica structure.

The TG curve (Fig. 3.1, red trace) exhibits three distinct mass loss regimes:

Low-Temperature Situation (< 400°C): Up to 400°C, beach sand samples have almost no mass gone less than an 2 weight percent), which is mainly due to water loosely attached and a little bit of volatile organic matter leaving[17]. The strong stability at this level shows these mature beach sands do not really pick up water and have not too much organic material since they went through serious weathering and sorting before.

Mid-Temperature Situation (400–600°C): Here we can observe medium amount of mass loss, about 18 weight percent and it is mostly around 512°C where a DTG maximum of -0.32 wt percent per minute is visible (shown in Fig. 3.2). This matches with an endothermic effect in the DSC (ΔH is 4.87 J/g, starting at about 450°C), which means clays remove hydroxyl groups, for example, kaolinite changing to other compounds plus water[16]. These clays, including something like an illite or even some smectite did not get filtered out, can often be seen in sand that comes from areas with river activity.

3.2 Thermal Behaviour of Red Sand (R -01)

Compared with clay-rich beach sand B-01, the red sand R-01, which comes from an inland ferruginous deposit in India, is showing a much greater thermal stability when at middle temperature range, as TG-DTG-DSC scans prove (as shown in Fig. 3.4). The sand has reddish color due to iron oxide impurities being present there[10]. When the sample is heated to 1200°C, about 79 percent weight is lost,and at 939°C, only 20.98 percent by mass remains showing cleaner silica matrix and that mainly the carbonates are breaking down at high temperature. The testing process was same as the B-01; nitrogen gas, heating speed of an 10°C each minute, nearly 20 mg sample in the alumina crucible.

The TG curve (Fig. 3.4, red trace) reveals exceptional stability below 800°C, followed by an abrupt and near-complete mass loss event. Low- to Mid-Temperature Region (less than 800°C): There is almost no mass decrease, less than an 1 weight percent can be noticed upto 800°C, which depicts there are not really any organics, clays which release water or water that absorbs from the air. DTG baseline in Fig. 3.5 has tiny variations meaning only occasional desorption events might occur around 200–400°C rates below minus 0.1 wt.% per min from a possible iron oxide surface moisture like the goethite or hematite. The DSC curve stays almost flat without major peaks showing the quartz-iron oxide system is very resistant.

High-Temperature Region (800 to 1100°C): There is huge loss in mass around 79 percent that happens quick from 800 to 1000°C, with center near 939°C (DTG is at its highest with − 0.60 wt.% per minute shown in Fig. 3.5). This matches with a strong endothermic peak in the DSC that starts at 820°C and is the most at 950°C. The ΔH is about 25–30 J/g which was got by integrating curve (Fig. 3.6). This is caused by an intensive taking away of CO_2 from lots of calcareous materials (for example, $CaCO_3$ turns into CaO and CO_2). The amount of mass lost is about 74 percent CO_2, so carbonate mineral is above 80% compared to non-siliceous part which is very high for red sandstones maybe due to getting more from diagenesis in dry places. Its very fast process (DTG peak high) means the calcite is so crystalline and diffusion difficulty is low.

When temperature goes higher than an 1000°C, TG graph stays stable at 20.98 wt.%.This shows the leftover quartz (SiO_2) and iron oxides that have become stable where no changes appear in inert atmosphere. The DTG graph in Fig. 3.5 kind of mostly has fluctuations at low temperature because of tool noise or less evaporation but a very symmetric and sharp peak at the high temperature is visible meaning one main breakdown occurs.

3.3 Thermal Behavior of River Sand (RS-01)

River sand RS-01 from kauvery not specified, but can be an showed much cleaner thermal qualities compared to both beach sand B-01 and the red sand R-01 which have more impurities according to the TG-DTG-DSC data (see Fig. 3.7 for the reference). Its overall mass loss is only about 9.17 percent up until 1200°C, while leftover mass reaches 90.83 percent at 1193°C. Quartz (SiO_2) is mainly present in this sand and small impurities result in some low-level activity[11]. The experiments used a nitrogen atmosphere, 10°C per minute heating rate,and small samples of around 20 mg inside the alumina containers, similar to before. It is observed that the heating rate was steady and controlled without any fluctuations. The nitrogen gas was flowing continuously through the system to maintain an inert environment. The behavior of the thermal degradation of this sample differs slightly compared to other sands in the study, you know. This shows importance of the purity of sample for thermal properties, especially when applications require heat resistance.This explaines that the results affirm the quality of the material for industrial use.

The TG curve (Fig. 3.7, red trace) delineates four subtle mass loss steps, underscoring the sample's homogeneity and low volatile content.

Very Low-Temperature Regime (< 100°C): There is small reduction in mass about an 3.42 wt.% with inflection observed at 84.9°C (DTG peak found near − 0.2 wt.%/min; Fig. 3.8), that is considered due to disappearing of the physisorbed and capillary water. This usually happens for river sands which keep the water at surface from water moving but do not have deeper hydration because of abrasion mechanical.

Low-Temperature Range (200–500°C): There is another loss of 2.04 percent by weight found near 470°C, which seems to show up with wide endothermic shoulder in the DSC curve that starts around 400°C, where the enthalpy comes out to between 5 and 7 J/g (as shown in Fig. 3.9). This may be a sign of

burning or removal of tiny organic particles, like humic leftovers from the riverside plants or possibly an small dehydroxylation in illite clays. Because the change is under 3% by weight it suggests a very low clay amount, possibly less than 5 percent in total. Mid-Temperature Area (500–600°C): A little drop of 3.75 percent by weight at 507.3°C matches a clear endothermic DSC peak, which is also at 507.3°C with ΔH of 29.67 J/g, that likely shows dehydroxylation of last clay pieces (like kaolinite turning into metakaolinite). The DTG peak rate gets up to -0.3 percent by weight per minute (look at Fig. 3.8), showing fast but not strong energy output as expected in scattered small-sized phyllosilicate minerals.

High-Temperature Condition (700–900°C): Biggest drop in mass, by 9.17 wt.%, takes place close to 781.4°C. The DTG reaches its peak at minus 0.4 wt.% per minute which connects to an wide endothermic change in the DSC starting near 750°C and peaking close to 795°C. The enthalpy change is around 120 J/g overall for the high temperature. This happens because some minor carbonates, such as calcite bits from the shells or the limestone, are breaking down, making almost 8.5 wt.% CO_2. The event is wide showing probably various particle sizes or maybe magnesium and calcium carbonates mixed, like the river alluvial sediments.

The DSC graph shows long high-temperature endotherm that has an peak close to 1294.5°C, and ΔH measures 120 J/g. This could mean like late decarbonation or maybe the quartz α-β inversion[20]. The α-β process is not so endothermic and it usually happens near 573°C, also it does not change mass.After 900°C the graph is steady at 90.83 weight percent which makes quartz main, with no sulfates or any refractory leftovers.DTG results (Fig. 3.8) reveal weak oscillations coming from sample purity, and symmetry in peaks confirms an first-order kinetic process.

3.4 Thermal Behavior of M-Sand (M-01)

Manufactured sand (M-Sand) M-01, made by a vertical shaft impactor crush from granite in southern Indian mining site, shows an engineered thermal resistance based on TG-DTG-DSC data (see Fig. 3.10). This man-made sample presents the least total mass loss, which is about 7.53 weight percent among all the materials tested. Leftover mass after heating to 1200°C ends at 92.47 weight percent, which comes from the high quartz content and almost no impurities formed in mechanical processing.The testing was done the same as before, using nitrogen gas, heating rate of 10°C per minute and using an alumina dish for ~ 20 mg samples.

The TG curve seen in Fig. 3.10 (the red color line) shows that almost nothing changes until it reaches 700°C, and after that there are little mass changes, which is evidence of sort of a balanced mineral content for the broken rock materials.

In Low-Temperature area (lower than 300°C), a slight dropping of mass at roughly an 0.45 weight percent happens supposedly at 752.7°C but a label says it appears before.Yet the graph shows it is steady in the beginning, only small amount lost (around 0.2–0.3 wt.%) beneath 200°C with a DTG minor peak of about minus 0.1 wt.%/minute in Fig. 3.11. This is caused by removal of an extra water from air or oils from making, which demonstrates less porosity of M-Sand when put near to the standard river materials.

Mid-Temperature Range (400–800°C): A small weight loss of 0.45 wt.% seen at 752.7°C matches with some weak endothermic movements in the DSC, beginning almost at 700°C and having ΔH below 5 J/g (Fig. 3.12). This might be linked to slight dehydroxylation in other phyllosilicates, like some mica remains or an altered feldspar substances, or a little organic material burning off. The DTG rate keeps low (-0.15 wt.% per minute at the highest), showing that less than two percent of the content reacts, which is common for processed aggregates.

High-Temperature Range (1000–1200°C): The main change here is a 0.58 wt.% mass loss at 1291.5°C (DTG peak hitting – 0.25 wt.% per minute; see Fig. 3.11), lining up with a strong DSC endothermic response at 1315°C (ΔH 79 J/g, partly calculated above 1200°C, shown in Fig. 3.12). This is probably caused by the leftover carbonate breaking down, like an unreacted calcite coming from limestone mixed into base rock, releasing carbon dioxide about 0.54 wt.%. The event's high temperature start and smaller (< 1 wt.%) level point out how the process efficiently separated volatile and unwanted materials.

The profile gradually goes to around 92.47 weight percent leftover at 1200°C, with an anhydrous silicates being major component, especially quartz above 90 weight percent. DTG shifts after 1000°C are because of changed heat capacity patterns not volatility. This high purity is much different from natural sand samples, such as B-01 having 65–70 percent lost, R-01 is losing 79 percent and RS-01 with 9.17%. Coats-Redfern method in isoconversional kinetics gives rather small activation energies, about 100 kJ/mol for middle temperature dehydroxylation, near 160 kJ/mol for decarbonation.

4. CONCLUTION

This work did a systematic look at thermal decomposition behaviors for four main types of sand: beach sand (B-01), red sand (R-01), river sand (RS-01) and sand that is manufactured (M-01). The experiment used the TG instrument with the DSC, heating to 1200 degrees Celsius. The main targets were to put numbers on mass losses, find where transitions between phases happened and show any energetic results and also gain decomposition kinetics. These steps help make comparisons if the sand is good to use for jobs needing high temperatures, like for building materials, sand used for mold foundries or in refractory setups. Results make it highly clear that differences in thermal stability come from mineral content, where the sands come from and how they have been processed. M-Sand is the sand with the best heat resistance, while river sand also comes next in the heat-resistant.

This study did systematic analysis on the thermal breakdown actions in four main sands. The tested sands are beach sand (B-01), red sand (R-01), sand from rivers (RS-01) and sand that is artificial (M-01). The experiment was by using TG together with DSC, where sands got heated up to 1200 degrees Celsius. The main plans were measuring how much mass is lost, trying to find temperatures at which different changes happen and also to provide energy data along with getting decomposition kinetic results. Doing these operations is helpful when wanting to check if any kind of sand is fit to use in situations where very high heat is involved, like construction usage, sand for foundry mold, or when using it for refractory situations. The outcomes completely reveal differences in thermal stability are meaningfully related to

where sand comes from, mineral parts, and production process. The heat resistance of M-Sand was mostly better, and the river sand also was a good performer.

Sands in general pass through many stages where they lose a mass and most of this loss, if checked, happens because of taking the water away first, then clays are decomposing and, after that, lime-type impurities start breaking apart. When all of these are tested under nitrogen, which is acting as a gas that does not react. The mass dropped in a large range, like 7.53 percent for a sample M-01 and up to 79 percent for one called R-01. This sequence features M-Sand as being much purer than the River Sand and River Sand is still cleaner than Beach Sand but the Red Sand carries most impurity stuff. The values for heat-up-taking, provided as integrated enthalpy (ΔH), got higher in ones with the biggest impurities, like R-01, which required around 25 to 30 Joules per gram during carbonate breakdown. On activation energy (E_an, according to Coats-Redfern), this one goes from about 100 kilojoules per mole for pure ones with easy steps, and almost 250 kilojoules per mole for R-01 with its harder carbonates. The peak for heat given out was not big except for little ones from quartz turning near 573°C for the RS-01, which supports the non-combustible burning of nitrogen.

Table 4.1 collects main differences in the samples and thermal data are also shown for easier judging. It can be seen that the river and the M-sand keep over 90 percent remaining, which is helpful to use under hot conditions without a major error. On the other hand, beach sand and red sand could create bloating or maybe gas emissions as it loses clay substances and CO_2 content during operation. The enthalpy recorded for the R-01 sample shows that possibly more detailed reactions are going on for it. This proves that lime contamination breakdown matters not only the water removal. In some of the samples, activation energy gives an idea of how stable a material is when it is heated. So the way heat goes into the material tells you about how good the sand is for each industry.

Table 4.1
Comparative Thermal Metrics of Sand Samples

Parameter	B-01	R-01	RS-01	R-01
Total Mass Loss (wt.%)	60–70	79	9.17	7.53
Residual Mass (wt.% at 1200°C)	33	21	91	92
Key Endotherm (°C, ΔH J/g)	512 (5); 904 (17)	939 (25–30)	507 (30); 795 (120)	1315 (79)
Dominant Loss Regime (°C)	500–1000	800–1000	200–900	1000–1200
E_a Decarbonation (kJ/mol)	220	250	180	160
Implied Quartz Content (%)	30–40	~ 20	> 90	> 90

Declarations

Funding:

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Consent to Publish Declaration:

Not applicable.

Ethics and Consent to Participate Declarations:

Not applicable.

Author Contribution

R.G wrote the manuscript text except conclusion andB.K prepared the table 4.1 and the Fig 3.1 -Fig 3.11T.D research all the results and wrote the conclusion.All the authors reviewed the manuscript.

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Figures

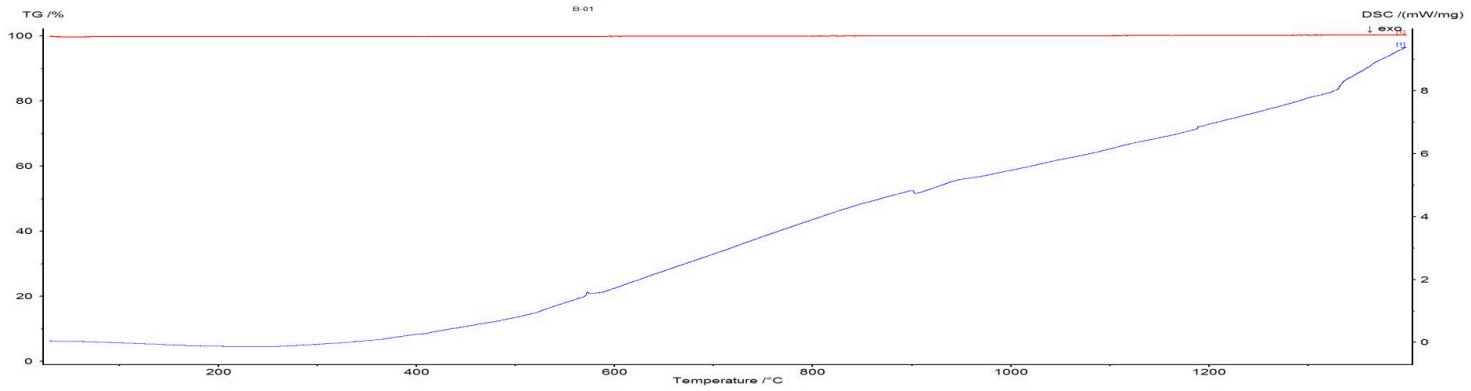


Figure 1

Combined TG-DSC curve for B-01, showing mass % (red line) and heat flow (blue line) versus temperature

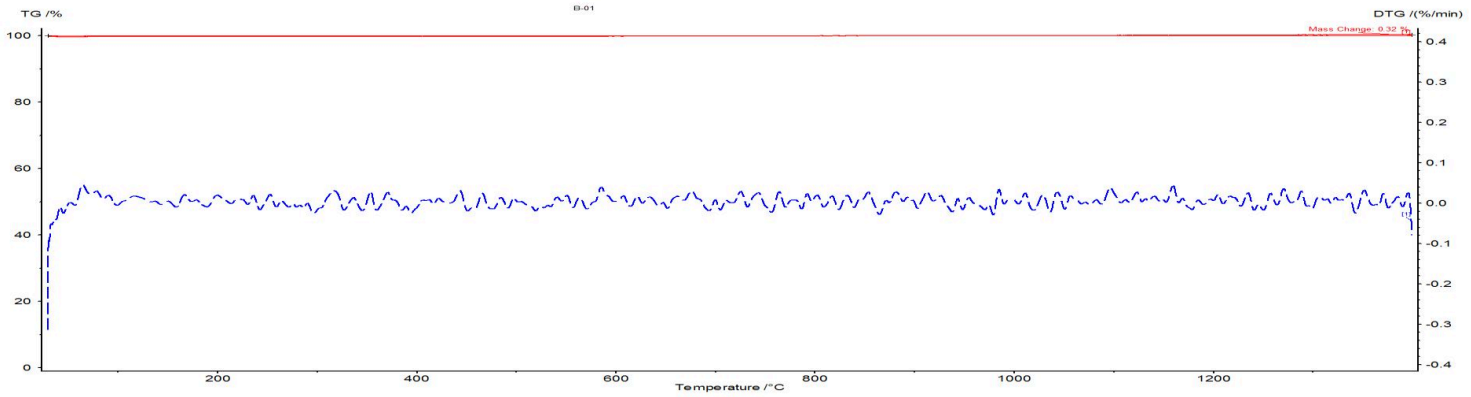


Figure 2

DTG curve for B-01, overlaid with TG, highlighting mass loss rates (%/min) on the secondary y-axis.

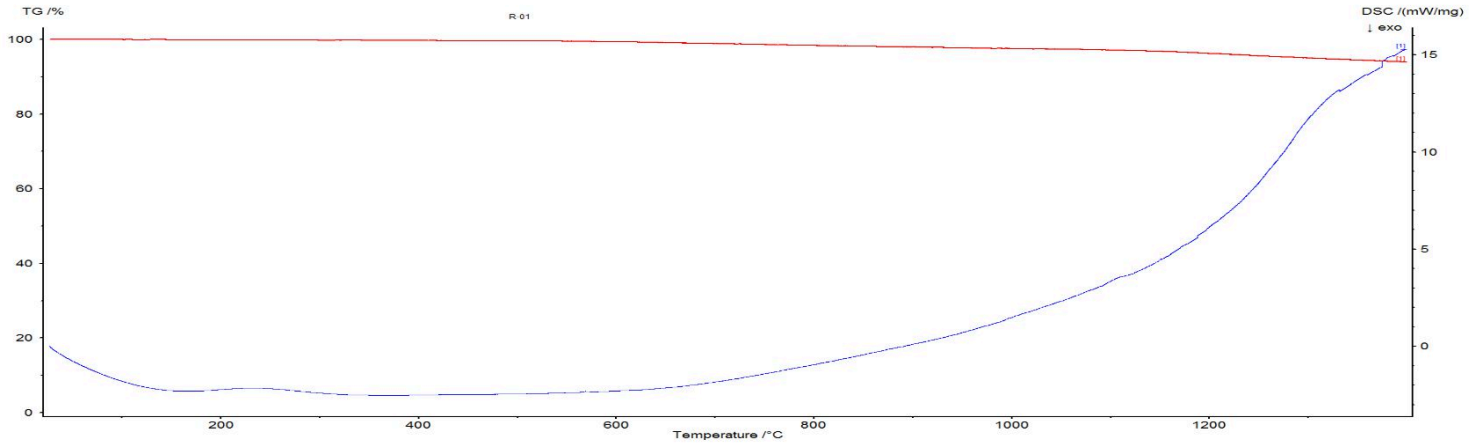


Figure 3

Combined TG-DSC curve for R-01

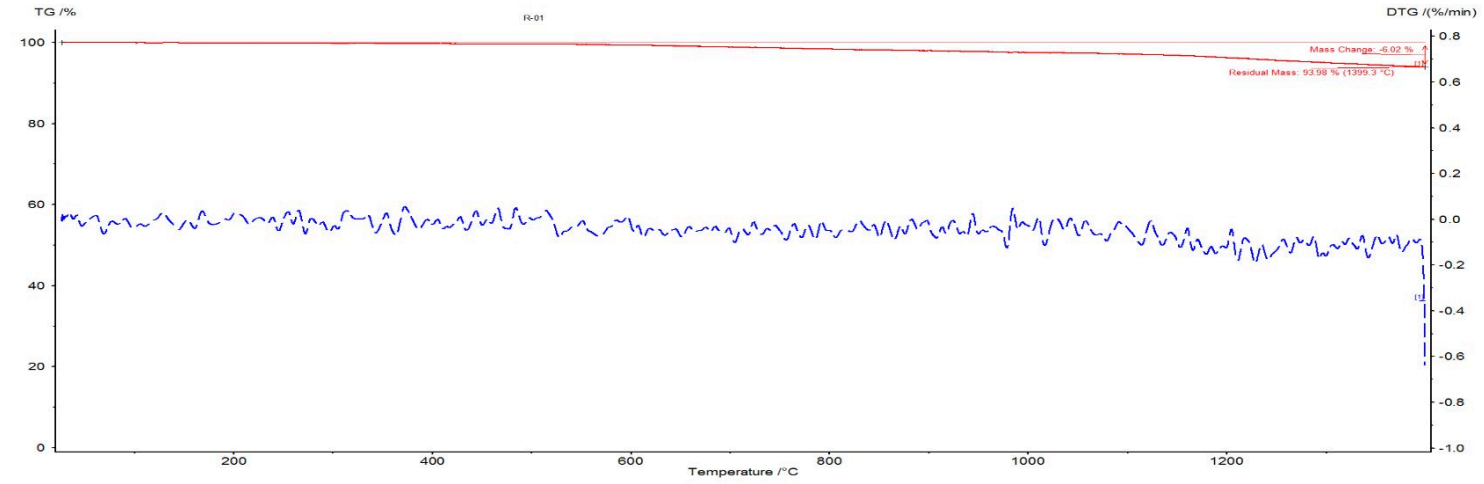


Figure 4

DTG curve for R-01, overlaid with TG

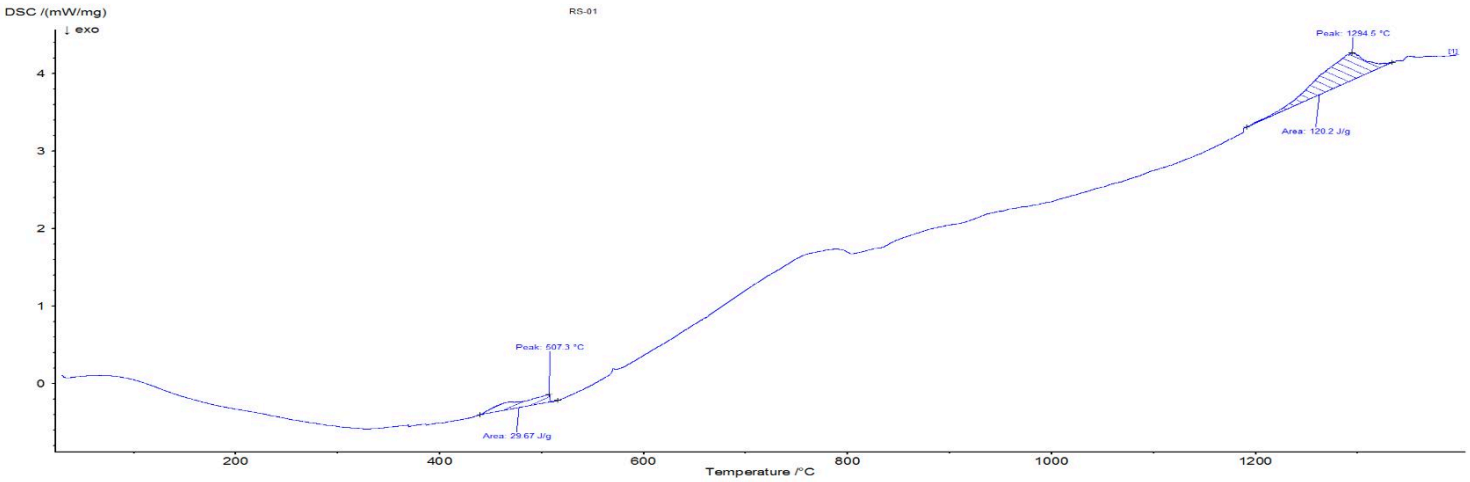


Figure 5

Standalone DSC curve for R-01

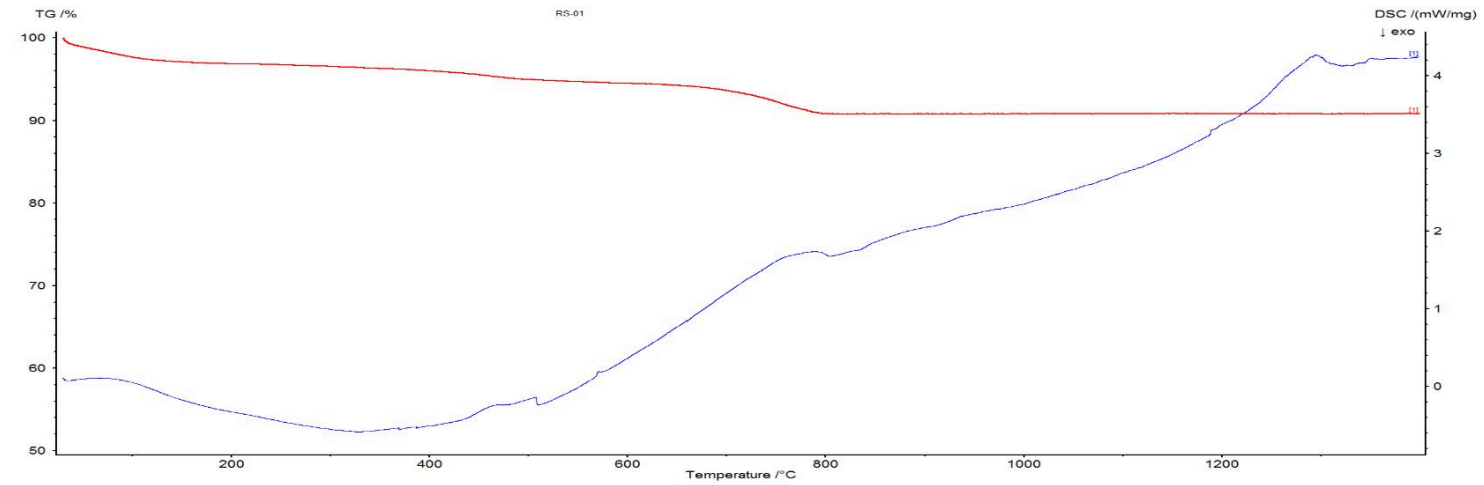


Figure 6

Combined TG-DSC curve for RS-01

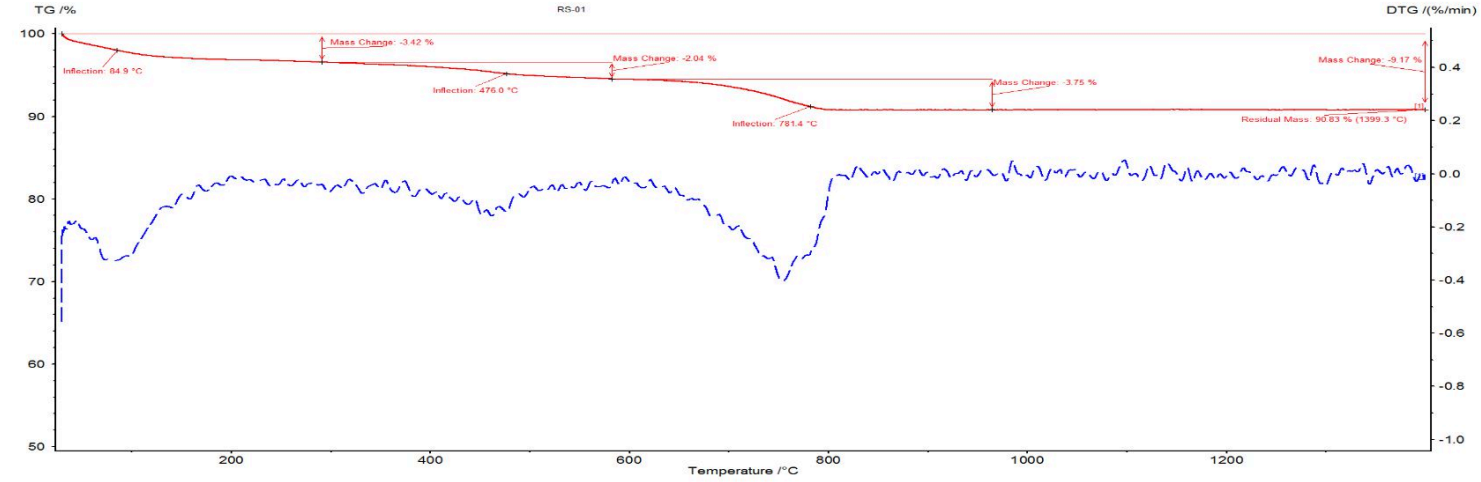


Figure 7

DTG curve for RS-01, overlaid with TG

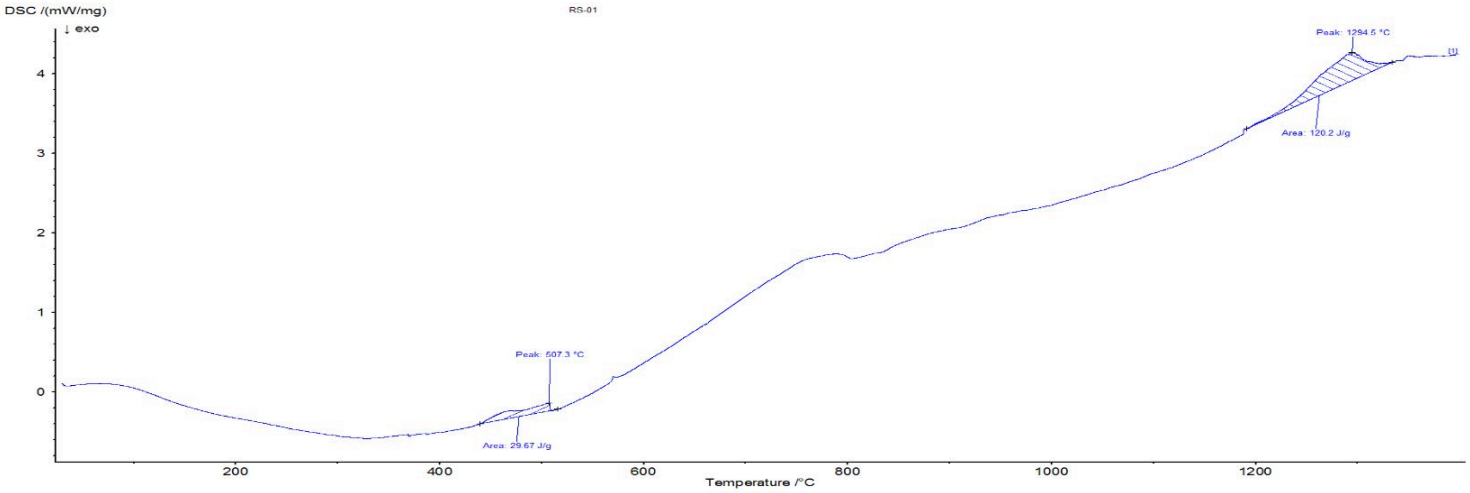


Figure 8

Standalone DSC curve for RS-01

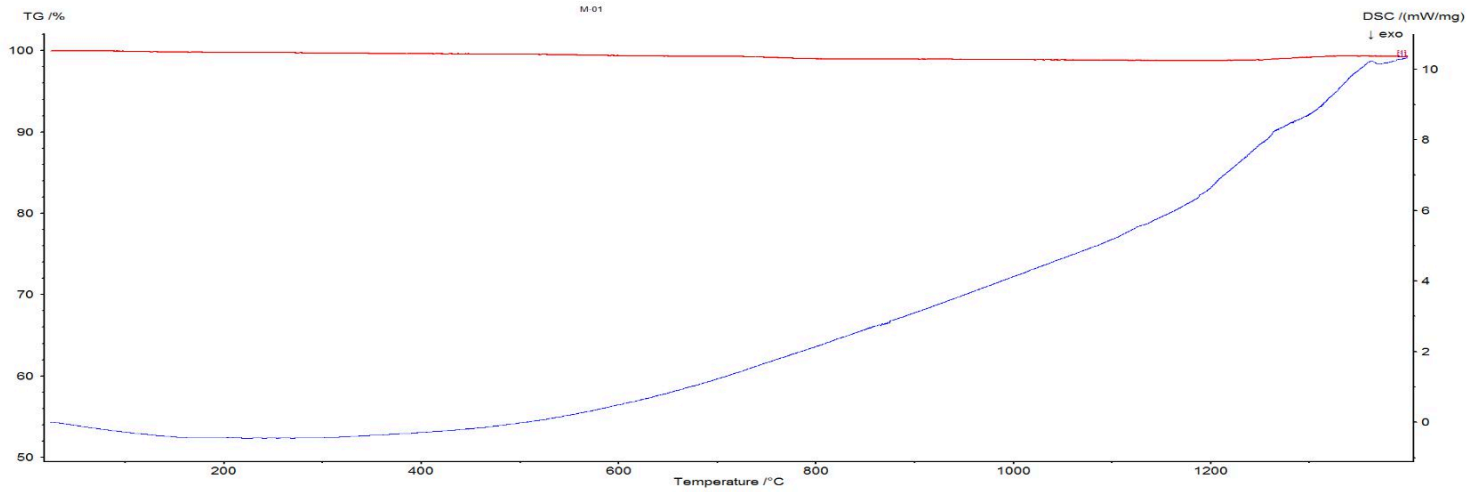


Figure 9

Combined TG-DSC curve for M-01

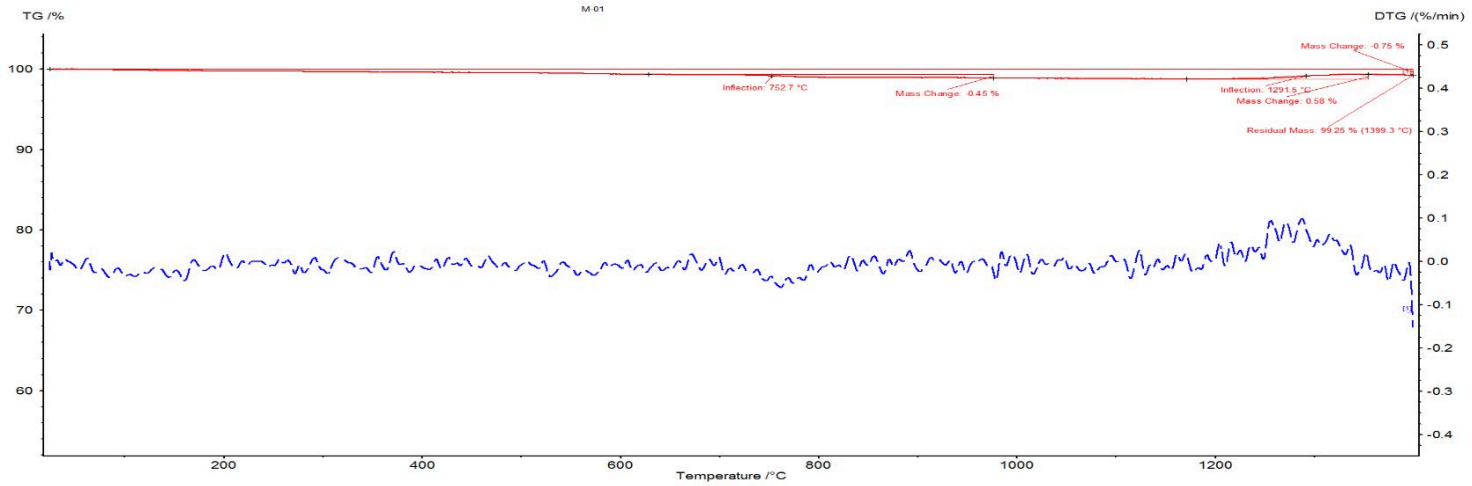


Figure 10

DTG curve for M-01, overlaid with TG

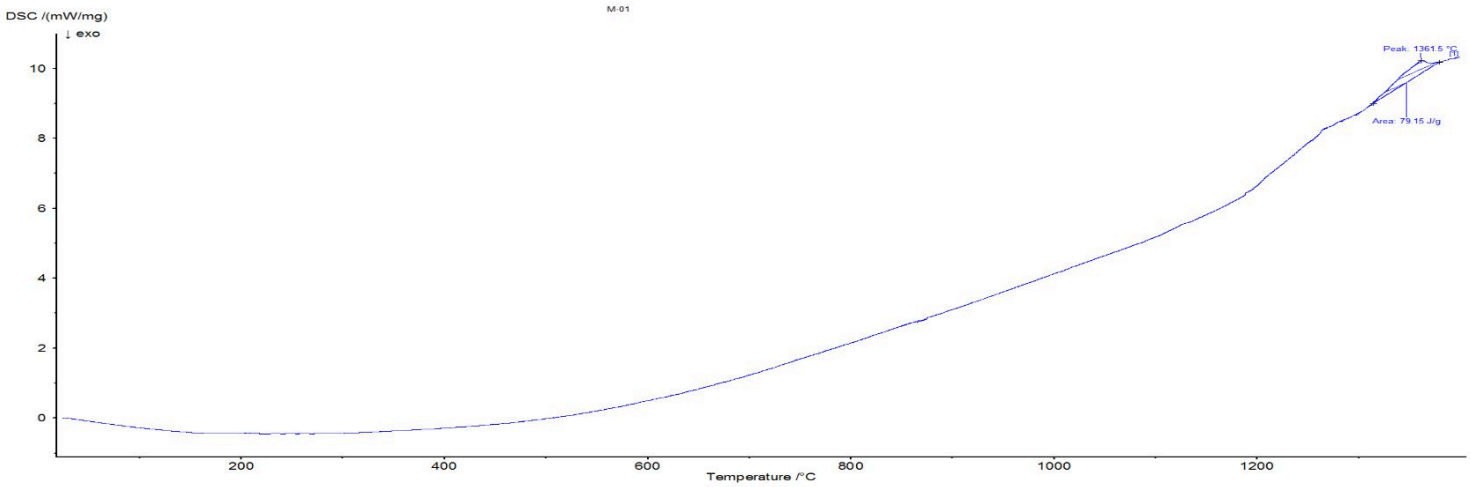


Figure 11

Standalone DSC curve for M-01