Intra-annual oxygen isotope variations in central Indian teak cellulose: possibility of improved resolution for past monsoon reconstruction

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A clear seasonal cycle has been detected in the intra-annual oxygen isotope variations (δ¹⁸O) of cellulose from several annual growth rings of teak trees that grew in central India. This persistent cycle is marked by higher δ¹⁸O values at the ring boundaries and lower δ¹⁸O values at intermediate parts. The amplitude of this seasonal cycle varies up to 6.8‰. Based on the pattern of teak growth reported in the literature and a plant physiological model that interprets the δ¹⁸O of plant cellulose, it seems possible to identify subsections of a ring that formed during pre-monsoon, peak-monsoon and post-monsoon. Comparison of the δ¹⁸O profile of a ring (year AD 1971), analysed with the highest resolution, and a model profile based on concurrent local meteorological data indicates the possibility of achieving a ~20 day resolution in monsoon reconstruction by intra-annual δ¹⁸O measurements.

Keywords: Intra-annual variations, teak, oxygen isotope.

CLIMATE of the earth is continuously changing due to natural as well as anthropogenic causes. For improving the prediction of future climate, it is imperative to quantify how climate changed in the past, especially during the past few centuries. To understand the climate of the pre-instrumental period, various proxies such as ice cores, lake sediments, corals, speleothems and tree rings are used. Among these, tree rings have specific advantages: they have a wide geographic distribution, are annually resolved, offer a continuous record, and are easily dated by ring-counting. Due to the absence of a pronounced seasonality in temperature, a factor responsible for growth rings in temperate regions, tropical trees rarely exhibit well-developed annual growth rings, teak (Tectona grandis) being an exception. It is distributed throughout tropical Asia, parts of Africa and Latin America and is ideally suited for quantifying past tropical climate variability.

Several studies show the potential of teak in reconstructing past climate. Ramesh et al. analysed hydrogen isotopic composition (δD) of cellulose of teak trees from Thane, India and found that the length of growing season controlled the δD variations. Jacoby and D’Arrigo analysed ring widths of teak trees from Java and showed that the growth was insensitive to the amount of wet season rainfall. Pumijumnong et al. showed the growth to be correlated with rainfall during the first half of the wet season. Buckley et al. reported the variability of teak growth in western Thailand to be correlated with rainfall during the beginning and end of the monsoon season. Borgaonkar et al. and Somaru Ram et al. based on analysis of teak trees from central and southern India, demonstrated significant correlations of ring-width variations with pre-monsoon and post-monsoon climate and suggested a moisture index rather than total rainfall as a major factor controlling ring-width variations.

In the studies mentioned here, whole ring properties of teak, e.g. ring-width and cellulose isotopic composition are used for reconstructing past climate by finding a response function which includes estimating regression coefficients between ring width/δD and seasonal rainfall of the concurrent year. More climatic information could be gleaned if one could divide each ring according to time (e.g. month) of its formation and then correlate its ring-width or isotopic composition with climatic parameters of the concurrent period.

Traditional techniques of extracting cellulose and measuring its isotopic composition have been laborious and time consuming leading to low sample throughput. Some recently suggested sample preparation techniques that ensure high throughput and have enabled processing smaller amounts of wood, thereby greatly facilitating intra-annual isotopic studies, hitherto not considered feasible. Recently developed plant physiological models have greatly improved the quantitative interpretation of the isotopic composition of plant cellulose.

Recent studies demonstrate the potential of intra-annual resolution isotope studies in deciphering past
climate. The applications of such studies range from the reconstruction of past rainfall, relative humidity, temperature, source water isotopic composition, tree growth rates to establishing chronometry in trees lacking discernible growth rings and to tracking tropical cyclones. In this article, we present the results of intra-annual oxygen isotopic composition ($\delta^{18}O$) studies in teak trees from central India. We compare the observed and modelled variations of intra-annual $\delta^{18}O$ to ascertain factors governing observed intra-annual $\delta^{18}O$ variations and possible time resolutions that can be achieved by intra-annual isotopic studies.

Materials and methods

Sampling

Several annual rings from three teak trees, two from Jagdalpur (lat. 19°05′N and long. 82°02′E, 560 m asl), Jag03 and Jag04, and one from Hanamkonda (lat. 18°01′N and long. 79°34′E, 224 m asl), AP1 were analysed for intra-annual $\delta^{18}O$ studies. Both the locations have India Meteorological Department (IMD) weather stations. Southwest monsoon (June to September) is the dominant source (>80% of the total rainfall) of rain in this region. The mean temperature (in °C) ranges from 19.7 (December) to 31.4 (May) for Jagdalpur and from 22.6 (December) to 34.3 (May) for Hanamkonda. July to September is the period of highest humidity, which decreases subsequently. Figure 1 shows the climatology of the two locations. Hyderabad (17.45°N, 78.47°E) is a Global Network of Isotope in Precipitation 26 (GNIP) station in the region, for which $\delta^{18}O$ of precipitation is available for a period from 1997 to 2001.

Disc samples were used in the present study. Upon polishing, the samples showed ring-porous structure. The dating of the samples was done employing standard tree-ring dating techniques and the dates were verified with other established teak chronologies from the same region.7,11. Radial strips along the selected directions were cut from the discs. To facilitate intra-annual sampling, wider rings were selected in general. Each ring was subdivided into four or higher segments along the radial directions using a scalpel and a chisel. The widest ring, corresponding to AD 1971, from one of the samples from Jagdalpur (Jag03), was sampled with the highest resolution; the ring was subdivided into 16 parts. While separating the segments, sufficient care was taken to avoid contamination from the adjacent segments. Figure 2 illustrates the scheme of sampling with the help of a ring subdivided into four segments. As cellulose is sequentially laid down in a ring, these segments correspond progressively to different phases of the growing season, and any averaging is likely restricted to a few days at most.

Cellulose extraction

The separated segments of the individual rings were powdered in a Wiley mill. $\alpha$-Cellulose was extracted from the powdered wood material using a method suggested by Gaudinski et al.14 with some modifications. The method of Gaudinski et al.14 called ‘MBrendel’ method, is a modification of the method of Brendel et al.17. About 50 mg of wood powder was taken for extracting cellulose. To ensure uniform heating, boiling of wood powder with acid mixture (step 1 of MBrendel) was done in an oven preheated to 120°C. In the present study, modifications were introduced in step 6 of the MBrendel method. These modifications are ultrasonication of the mixture (sample and NaOH) for ~5 min and keeping the solution for 1 h instead of ~10 min as suggested in the original method. The ultrasonication was introduced to disintegrate sample pellets and facilitate reaction with NaOH. In a day, cellulose was extracted from two batches of samples each containing 16 samples, a number determined by the capacity of the centrifuge.
The extracted cellulose was whitish in colour. Purity of the extracted $\alpha$-cellulose was checked by Fourier Transform Infrared Spectroscopy (FTIR) using a Varian 3100 FTIR spectrometer at the Ahmedabad Textile Research Institute (ATIRA), Ahmedabad, India. All the measurements were done using KBr pellets. Figure 3 shows representative spectra of $\alpha$-cellulose extracted by the methods used in the present study and given by Brendel et al.\textsuperscript{12}. Also shown in the figure is a spectrum of the commercially available $\alpha$-cellulose from Sigma Aldrich. The absorbance spectra of each sample shown in Figure 3 have been normalized by the absorbance of the highest peak of the individual samples. Anchukaitis \textit{et al.}\textsuperscript{27} reported that the cellulose extracted by the methods of Brendel \textit{et al.}\textsuperscript{12} and MBrendel resulted in acetylation of cellulose with its peak in FTIR spectra at 1720 cm\textsuperscript{-1}. They further cautioned that acetylation introduces a ‘dead’ carbon in the cellulose structure which interferes with carbon isotope measurements, especially radiocarbon (C\textsuperscript{14}). It can be seen from the FTIR spectra in Figure 3 that the $\alpha$-cellulose extracted in the present study does not show the acetylation peak, perhaps indicating complete deacetylation of cellulose by saponification with NaOH.

\textbf{Isotopic measurements}

The isotopic measurements were done using Thermo Quest’s Finnigan Delta plus continuous flow Isotope Ratio Mass Spectrometer (IRMS) at the National Facility, University of Agricultural Sciences, Bengaluru, India. The peripherals attached with the mass spectrometer were High Temperature Conversion Elemental Analyser (TC/EA) and ConFlo III.

For isotopic measurements, about 0.85 mg of cellulose was packed in silver foil and the sample capsules were kept at 60°C for at least 10 h before measurements (to ensure removal of adsorbed water vapour, which might interfere with the oxygen isotope analyses). Typically, 50 samples were analysed in a single run. These contained 44 cellulose samples and 6 standards with standards at 1st, 10th, 20th, 30th, 40th and 50th positions. The standards used were in-house calibrated starch ($\delta^{18}$O = 26.8‰) and Australian National University (ANU) sucrose ($\delta^{18}$O = 36.4‰). TC/EA was operated at 1350°C to ensure complete pyrolysis of the samples. To avoid isotopic interference of CO and N\textsubscript{2}, the pyrolysed gases were passed through gas chromatograph (GC) column. All the measurements were done with ConFlo III on ‘Helium dilution ON’ mode. During the isotopic measurement of a sample, three reference gas pulses were injected in IRMS, which is followed by a sample gas injection followed by another reference gas injection. Time required for the measurement of one sample was 10 min. The reference gas injections gave an internal precision better than 0.1‰. The external precision of the measurements was consistently better than 0.3‰ ($N = 127$). Typically the reactor was changed after every 250 samples. The system was degassed overnight with TC/EA at 1350°C and GC at 300°C after changing the reactor. The isotopic values are reported relative to VSMOW as $\delta^{18}$O (‰), where $\delta^{18}$O = $\left\{ \frac{R_{\text{sample}}}{R_{\text{standard}}} - 1 \right\} \times 1000; R = ^{18}\text{O}^{16}\text{O}$.

\textbf{Models for interpreting $\delta^{18}$O of plant cellulose}

\textit{Isotopic composition of tree cellulose}

It is well understood that the $\delta^{18}$O of plant cellulose is influenced by climatic and biochemical processes. $\delta^{18}$O of the source water, $\delta^{18}$O of atmospheric vapour, the
level of evaporative enrichment of the water in the leaf during transpiration, biochemical fractionation during sucrose synthesis in the leaf and the extent of exchange between sucrose and xylem source water during synthesis of cellulose are important in deciding $\delta^{18}O$ of plant cellulose.

Modifying the Craig and Gordon model\textsuperscript{28}, describing the isotopic fractionation during evaporation from open water bodies, by incorporating leaf boundary layer effects and diffusion through stomata, Dongmann et al.\textsuperscript{29} and Flanagan et al.\textsuperscript{16} gave a model for isotopic composition of the leaf water ($R_{wl}$).

\[
R_{wl} = \alpha^* \left[ \alpha_k R_{wx} \left( \frac{e_1 - e_a}{e_1} \right) \right] \\
+ \alpha_{kb} R_{wx} \left( \frac{e_a - e_1}{e_1} \right) + R_s \left( \frac{e_a}{e_1} \right),
\]

(1)

where $R_{wl}$, $R_{wx}$ and $R_s$ refer to the oxygen isotopic ratio ($^{18}O/^{16}O$) of leaf water, xylem water and bulk air respectively; water vapour pressure of intercellular leaf space is $e_i$, of leaf surface is $e_v$ and of bulk air is $e_a$; $\alpha^*$, $\alpha_k$, and $\alpha_{kb}$ are the liquid–vapour equilibrium fractionation factor, kinetic fractionation factor and kinetic fractionation factor associated with leaf boundary layer respectively.

Sucrose formed by photosynthesis in the leaf carries the isotopic composition of the leaf water ($R_{wl}$) along with an enrichment of $\sim 27\%$. In mechanistic model\textsuperscript{15} for interpreting $\delta^{18}O$ of tree cellulose, the final isotopic composition of tree cellulose ($\delta^{18}O_{cx}$) is given by:

\[
\delta^{18}O_{cx} = f_o (\delta^{18}O_{wx} + e_o) + (1 - f_o) (\delta^{18}O_{wl} + e_o),
\]

(2)

where $f_o$ is the fraction of carbon-bound oxygen that undergoes exchange with medium water, $\delta^{18}O_{wx}$ is xylem water, $\delta^{18}O_{wl}$ refers to oxygen isotopic composition of the leaf water at the site of sucrose synthesis and $e_o$ is the biochemical fractionation factor ($\sim 27\%$).

It can be seen from the eqs (1) and (2) that the $e_o/e_1$ ratio and hence relative humidity plays a crucial role in deciding the isotopic composition of leaf water and the subsequently synthesized cellulose\textsuperscript{30}; lower the relative humidity, more enriched would be the $\delta^{18}O$ of synthesized cellulose. In tropical areas such as central India, relative humidity varies (Figure 1) enough during the growing season to leave its imprint on the isotopic composition of cellulose\textsuperscript{31}.

Farquhar and Lloyd\textsuperscript{32} showed that the isotopic composition of the leaf water calculated using eq. (1) is more enriched in $^{18}O$ than that of the bulk leaf water and explained it in terms of the Péclet effect – transpirational advection of $^{18}O$ depleted (xylem) water to the evaporating site opposed by backward diffusion of $^{18}O$ enriched water into the leaf. Barbour et al.\textsuperscript{19} have proposed a model that accounts for the Péclet effect. To use this model, Péclet number for teak trees is required. This involves the measurement of effective path length, a model parameter that accounts for the discrepancy between the $\delta^{18}O$ values predicted by the Craig–Gordon model and measured bulk leaf water measurements. As an estimate of this parameter is unavailable, we used a model proposed by Roden et al.\textsuperscript{15} for calculating the cellulose $\delta^{18}O$, keeping in mind that the values so obtained could be higher in $\delta^{18}O$ than the actual. As the discussion here is based on relative variation, this should not hamper our interpretations seriously.

Modelling of intra-annual teak cellulose

The model\textsuperscript{15} available at http://ecophys.biology.utah.edu/public/Tree_Ring/ was used to construct the $\delta^{18}O$ profile of tree cellulose synthesized during the growing season, assuming no intra-seasonal mixing of photosynthates. The input parameters used in this model and their sources are shown in Table 1.

Stomatal conductance values for different months/days were estimated based on a relationship between vapour pressure deficit and stomatal conductance shown by Kallarackal and Somen\textsuperscript{33}. Mean monthly climatological data of temperature, relative humidity and station level pressure were taken from climatological tables published by India Meteorological Department\textsuperscript{34}. These values are means of monthly observation for a period from 1951 to 1980. Daily weather data used for constructing intra-annual $\delta^{18}O$ profile for year AD 1971 were taken from IMD’s Indian Daily Weather Records (IDWR) reports\textsuperscript{35}.

The model given by Roden et al.\textsuperscript{15} is mostly sensitive to relative humidity, $\delta^{18}O$ of source water and $\delta^{18}O$ of atmospheric water vapour. Figure 4 depicts the mean climatological intra-annual $\delta^{18}O$ profile (profile marked by ‘star’) and those obtained keeping one of the input parameters constant at the mean of all the monthly mean values. Each data point in climatological $\delta^{18}O$ profile is obtained from considering mean of the monthly observations for the period AD 1951–1980. For these profiles, a constant value of $\delta^{18}O$ of rainwater ($\sim 2.7\%$) and atmospheric water vapour (–12.5%) is used. The figure also shows climatological $\delta^{18}O$ profile when $\delta^{18}O$ of rain water and atmospheric vapour are changed respectively from –2.7 to 0.0% (profile marked by ‘open triangle’) and –12.5 to –15.0% (profile marked by ‘filled triangle’). The former systematically offsets the climatological profile by its amount of change while the effect of the latter is seen more during June to September, period of higher relative humidity. The higher isotopic exchange between the leaf/stomatal water and atmospheric water...
Table 1. Climatic and isotopic input parameters used in the model for calculation of cellulose $\delta^{18}O$ values and their sources

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Source</th>
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<tbody>
<tr>
<td>Source water/precipitation $\delta^{18}O$ (%)</td>
<td>GNIP$^{15}$ station: Salagiri</td>
</tr>
<tr>
<td>Atmospheric water $\delta^{18}O$ (%)</td>
<td>Considered 11‰ depleted than $\delta^{18}O$ of rainfall</td>
</tr>
<tr>
<td>Medium water $\delta^{18}O$ (%)</td>
<td>Considered the same as that of the source water</td>
</tr>
<tr>
<td>Relative humidity (%)</td>
<td>Climatological tables$^{36}$/IMD IDWR$^{37}$</td>
</tr>
<tr>
<td>Air temperature (°C)</td>
<td>Climatological tables$^{36}$/IMD IDWR$^{37}$</td>
</tr>
<tr>
<td>Leaf temperature (°C)</td>
<td>Linacre$^{38}$</td>
</tr>
<tr>
<td>Barometric pressure (kPa)</td>
<td>Climatological tables$^{36}$/IMD IDWR$^{37}$</td>
</tr>
<tr>
<td>Stomatal conductance (mol m$^{-2}$ s$^{-1}$)</td>
<td>Kallarackal and Somen$^{35}$</td>
</tr>
<tr>
<td>Boundary layer conductance (mol m$^{-2}$ s$^{-1}$)</td>
<td>Grace et al.$^{39}$</td>
</tr>
</tbody>
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vapour during the period of higher relative humidity lowers the leaf water $\delta^{18}O$ and hence that of the synthesized cellulose. It can be seen clearly from Figure 4 that (i) the overall shape of the intra-annual $\delta^{18}O$ variations could be explained mainly by variations in the relative humidity; the seasonal amplitude is clearly lowered when humidity is kept constant, (ii) cellulose synthesized during the peak monsoon season (July, August and September) has the lowest $\delta^{18}O$ values compared to those synthesized during pre- and post-monsoon seasons, (iii) relative humidity, $\delta^{18}O$ of source water and $\delta^{18}O$ of atmospheric water vapour are the main parameters influencing intra-annual $\delta^{18}O$ values.

Growth pattern of teak

Relationship between rainfall and teak growth was studied by Sudheendrakumar et al.$^{36}$, Priya and Bhat$^{37}$ and Buckely et al.$^{38}$. Sudheendrakumar et al.$^{36}$ have observed a bell-shaped teak growth curve with higher growth rates during the months of higher rainfall. Cambial activity studies by Priya and Bhat$^{37}$ have shown that the pre-monsoon showers break the cambial dormancy and higher amount of rainfall contributes to the greater amount of wood formation. The authors also pointed out concurrence of the period of the highest cambial activity and the period of the highest rainfall.

Results and discussion

The mean climatological intra-annual $\delta^{18}O$ profile shown in Figure 5a is estimated by the model$^{15}$. The Monte Carlo method was used to vary the input climatic parameters. Monthly $\delta^{18}O$ values and associated 1-sigma uncertainty are derived from 1000 model runs with simultaneous and random 1-sigma perturbations with normal distribution of the input parameters. 1-Sigma uncertainty in the model profile shown in Figure 5a is based on 1-sigma standard deviations of result of 1000 model runs. The modelled profile is constructed considering constant value of $\delta^{18}O$ of rainwater.

The monthly data points in the modelled profiles in Figures 4 and 5a are interpreted as follows: if enough moisture is available in the soil for teak to grow, it would respond to the ambient climatic parameters and would lay down wood whose cellulose $\delta^{18}O$ values would be as per the data points in the profiles. Teak being a deciduous tree, does not grow throughout the year. Dearth of soil moisture, especially during January to May, leads to very little diameter growth. Further, the amount of wood formed during different months is variable (discussed earlier). Although the intra-annual segments (starting from the pith side) represent the general progression of time, they do not represent equal periods of time. Consequently, months when the diameter growth is less are under-represented in the observed intra-annual $\delta^{18}O$ profile.

One of the new results obtained in this study is that the observed intra-annual $\delta^{18}O$ variations of teak show a typical pattern with lower $\delta^{18}O$ values in the middle and higher values at the ring boundaries. The amplitude, difference between the lowest and the highest $\delta^{18}O$ values...
of different segments of a ring, vary respectively from 3.0 to 6.8‰ and from 1.9 to 5.0‰ in fine and coarse resolution analyses. Figure 5b presents the typically observed intra-annual δ18O profile, mean of the profiles of annual rings of teak from Jagdalpur (2 trees; 6 rings) and Hanamkonda (5 rings). The error bars associated with the mean profile show typical segment-wise spread in the intra-annual δ18O values. Based on the pattern of teak growth, an approximate period can be assigned to the various segments (top axis of Figure 5b). The figure also presents the modelled intra-annual δ18O profile along with the 1-sigma uncertainty (shaded area), constructed based on the profile in Figure 5a and the growth pattern of teak. The points from the pith side in the shaded area are respectively the means of monthly modelled values of May–June, July–August, August–September and October–November–December. The observed δ18O values are less than the modelled possibly because of the Péclet effect (discussed earlier). It is further observed that the spread in δ18O values at the intermediate portion is more in the observed profile than in the modelled profile. One of the possible reasons could be changes in δ18O values of rainwater during July to September, which is unlikely formed during the periods of lower relative humidity and is likely formed during pre-monsoon (part near the pith side) and post-monsoon (part near the bark side) times. The intermediate part characterized by lower δ18O values corresponds to growth that took place during main-monsoon season.

Ring of AD 1971 in Jag03 was the widest (15 mm) and was subdivided into 16 parts. Figure 6 shows a comparison of the actual δ18O profile of this ring (Figure 6a) with the profile constructed using the model15 (Figure 6b). The expected δ18O values of cellulose that formed daily during AD 1971 were calculated using the daily local meteorological data of the same year at Jagdalpur. The constructed daily profile and its 20-day running mean are depicted in Figure 6b. Observed daily variations in relative humidity (thin line) and rainfall (bars) at Jagdalpur during AD 1971 are shown in Figure 6c.

Assignment of precise time intervals to the data points in Figure 6a is difficult as the times of initiation and cessation of radial growth and variation of the growth rate are only roughly known. Nevertheless, based on general observations regarding growth of teak – a month’s interval between bud break and initiation of radial growth37 – the first segment can be assigned to mid-May. The last segment could possibly represent the end of November as leaf fall starts about a month after the last rain39 and growth rate decreases rapidly afterwards. The comparison of observed and modelled intra-annual δ18O profiles in Figure 6 is striking. The actual intra-annual δ18O profile (Figure 6a) and the modelled profile with 20-day running mean (Figure 6b) for the corresponding duration (mid-May to the end of November) show similarities in trends and amplitudes. This attests to the efficacy of plant physiological models in interpreting the observed intra-annual δ18O variations in teak trees.

Intra-annual isotope studies of trees can shed light on the possible time resolution that can be achieved by isotope dendroclimatological investigations, which is a function of the sampling resolution and the extent of mixing and isotopic averaging of photosynthates. Kangawa et al.40 using carbon isotope (δ13C) tracer in Cryptomeria japonica tree have suggested an achievable time resolution of 8.7–28 and 33–42 days for the early wood and late wood respectively. Another way of addressing this issue is the comparison of the observed and modelled intra-annual δ18O profiles. Visual similarity of the observed intra-annual δ18O profile (Figure 6a) and modelled profile with the 20-days running mean (Figure 6b) suggests the possibility of achieving ~20 day of resolution during the peak growing season (June–September).

As Ramesh et al.4 have earlier demonstrated the coherence of isotopic variations among different teak trees
growing in the same region, our results are likely to hold for other teak trees growing in central India.

Conclusions

We demonstrate that teak trees from central India show a very clear seasonal variation in the intra-annual cellulose $\delta^{18}O$ year after year. The identification of the seasonal cycle induced by relative humidity and $\delta^{18}O$ of rainfall is useful in establishing chronology in other tropical trees lacking discernable growth rings, following the approach of ‘tropical isotope dendrochronology’ suggested by Evans and Schrag:13; wood corresponding to one seasonal cycle of $\delta^{18}O$ is considered as a ‘ring’ and regular dating/counting methods are used to assign calendar years to the tropical trees lacking visible growth rings.

Based on the observed pattern of intra-annual $\delta^{18}O$ variations each ring can be divided into three subsections: one closer to the pith side with higher $\delta^{18}O$ values; the second with lower $\delta^{18}O$ values in the middle; the third towards the bark side with higher $\delta^{18}O$ values. These subsections roughly correspond to pre-, main- and post-monsoon periods respectively. As these parts preserve time averaged ambient environmental conditions, their $\delta^{18}O$ values could be used to decipher climatic conditions during respective times. It seems possible to have a time resolution of ~20 days by intra-annual studies.

1. Tiwari, M., Managave, S. R., Yadava, M. G. and Ramesh, R., Spatial and temporal coherence of paleomonsoon records from marine


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