Real-world Shelf-Life Study of Tissue Equivalent Liquids for SAR evaluation

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Abstract

SAR assessment, as defined by the IEEE-1528 standard, has been the standard for defining the safe exposure of the RF radiations and usage limits for wireless devices. The Tissue Equivalent Liquids (TELs) are essential parts of the SAR assessment system, as they mimic the human body's dielectric properties at their specific frequencies and allow for testing of the range of the wireless devices as per specified procedures. Since, the TELs are artificially synthesized compositions, they also need to meet the regulatory requirements of labelling the product and undergo shelf-life assessment for estimating the usable product life. In this paper, we present the real-world shelf-life study of few TEL compositions of different frequency range. The dielectric properties of these TELs at their respective frequency values/range are assessed and compared for over a period of 18 months, at different intervals, while keeping the TELs stored as per IEEE specified storage conditions i.e. temperature between 18°C and 25 °C (within ±1 °C) with RH between 40%-60%.

Keywords: Tissue Equivalent Liquid (TEL); IEEE-1528; Specific Absorption rate (SAR); Shelf-life study.

1. Introduction

Due to rapidly increasing usage of mobile phones, wireless devices, wireless local area networks (WLANs) and other IoT (Internet of Things) devices; exposure of radio and microwave frequencies has become part of our daily life and is increasing day by day. RF exposure and safety concerns about usage of wireless devices have been getting increased attention in recent years. One of the key components of RF exposure evaluation is the SAR (Specific Absorption Rate) measurement, which is a measure of the heating value of radiated RF energy on human tissue. SAR evaluates the relative safety of low power transmitters in close proximity to the human body or high-power transmitters at greater distances. SAR assessment is carried out, as per procedures defined in IEEE-1528 [1, 2] and has been the standard for evaluating the safe exposure of the RF radiations and usage limits for wireless devices. Various national and international regulatory agencies have made it compulsory to test the SAR-compliance of each wireless handheld device with the standard norms such as IEEE Std C95.1 [3] & ICNIRP guidelines [4].

Phantom models, simulating the human anatomies (head, body), are used for assessing the SAR, rather than carrying out whole assessment on a living human. These phantom models make use of tissue equivalent materials having similar properties as human tissues at specified frequencies. These tissue equivalent materials can be solid, liquid or gel type [5]. In our earlier work [6], we have developed formulae/recipes for development for frequency specific Tissue Equivalent Liquids (TELs) for 2G, 3G, 4G and WiFi frequencies (between 834 MHz to 2450 MHz). These recipes are based on pre-determined ratio of their respective constituents. Since, these TELs are essentially compositions of chemicals, they are also required to meet with the regulatory requirements of product labelling (expiry date, best before date, etc.). This is where, the determination of product "Shelf-life", given as the length of time a product can be stored, shelved and displayed, whilst still maintaining an acceptable quality or specific functionality [7], becomes important for these TELs.

Since, for TELs, only the dielectric properties (i.e. dielectric constant (ε_r') and electrical conductivity(σ)) are the acceptability criteria, we analyse the dielectric properties of these TELs for accelerated and real-world conditions over a period of 18 months and present them here. A tolerance range of ±5% for variation in dielectric properties (permittivity & conductivity both) is prescribed by the standard, hence same has been used as acceptance criteria for this study.

2. Methodology

The IEEE-1528 standard document (Annexure - C) [1] has provided details of various materials which can be used for preparation of TELS, along with suggestive composition ratios. For the present work, we have used following two types of compositions:

(i) Comp_A: Composition of De-Ionized water (min. resistivity 1 MΩcm), Diethylene glycol butyl ether (DGBE) (>99% pure) and Sodium Chloride (NaCl) (> 99% pure) &

(ii) Comp_B: Composition of De-Ionized water (min. resistivity 1 MΩcm), Diethylene glycol butyl ether

(DGBE) (>99% pure), Polyethylene glycol mono [4-(1,1,3,3-tetramethylbutyl) phenyl ether] (common name Triton-X) and Sucrose (> 98% pure)

Throughout this study, this nomenclature would be used. The ratio of the constituents in these compositions have been predetermined and available in our previous work [6].

2.1 Dielectric Property analysis

The "Open-ended Coaxial Probe" method is the most commonly used method for dielectric assessment of liquids due to its ease of measurement and simplicity. We have used the open-ended coaxial probe method based Liquid Dielectric assessment facility available at CSIR-National Physical Laboratory (NPL), which utilizes a commercial dielectric assessment system comprising of DAK 3.5 sensor probe (Make: S. P. E. Ag) and a Vector Network Analyzer (ZNB 8, Make: R&S), along with commercial software package DAK 2.4.0.814. Further details of the method and procedure adopted can be found in [8].

2.2 Shelf-Life Testing

The procedure for assessment of shelf life for products have been widely studied and standardized worldwide. The shelf life for TELs can be assessed using the procedures specified by ISO standard 16779:2015 [9] and ICH guidelines [10], for their dielectric properties Minimum data of three batches are required to estimate the shelf life. The IEEE-1528 standard has specified the storage conditions for TELs as temperature between 18°C and 25°C (within ±1 °C) with RH between 40%-60%.

Firstly, accelerated condition study is to be carried out. For items specified to be stored at room temperatures (like TELs), the long-term storage assessment should start from occurrence of significant change in product stored in accelerated conditions. If no significant change is observed in accelerated conditions, then long-term storage conditions data determine the shelf life. If the long term and conditions data show very little change or no change at all then it might be assumed that product would be stable during proposed shelf-life period. If long term or accelerated condition data shows change then statistical analysis determines the shelf-life.

Hence, the present study has been carried out in following two parts:

(i) Accelerated Shelf-Life Study

Three set of samples are kept from each of TEL compositions. First sample set (Comp_A_1 & Comp_B_1) is kept at normal storage conditions for entire duration. The second sample set (Comp_A_2 & Comp_B_2) is kept at elevated temperature (45°C) and third sample set (Comp_A_3 & Comp_B_3) is kept at lower temperature (8°C) for 10 days period each. Measurements are taken at normal temperature (i.e. 22°C ±1 °C), at the start (i.e. day 1) and after completion of 10 days in these conditions. After measurements on 11th day, second sample set is kept at lower temperature and third sample set is kept at elevated temperature for another 10 days and measurements are taken for all samples again, after completion of 20th day.

(ii) Real-world shelf-life study

One set of samples from the TEL compositions (Comp_A_4 & Comp_B_4) are kept stored at the specified conditions (i.e. between 18°C and 25°C (within ±1 °C) with RH between 40%-60%) for a period of 18 months and measurements are taken at start, completion of 3rd, 12th and 18th month at normal temperature (i.e. 22°C ±1 °C).

3. Results & Discussion

3.1 Accelerated Shelf-life Study

The study of dielectric properties for the TEL compositions under accelerated conditions has been shown in figure 1 and figure 2. Figure 1(a) shows the comparison of the three sample sets for composition A for 10 days under accelerated conditions, while figure 1(b) shows results for 20 days. The sample sets are exposed to accelerated conditions as discussed previously. The 5% tolerance band is also shown in the figure.

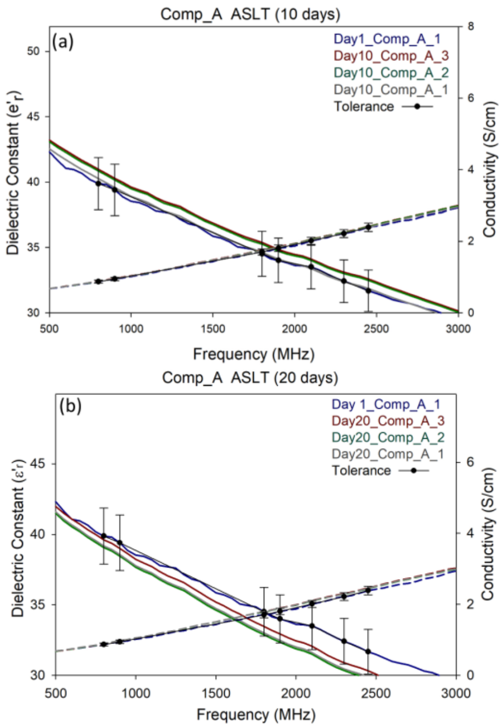

Figure 1. Comparison of dielectric properties of composition A at start of test (Comp_A_1 Dark blue) and (a) Samples after 10 days in accelerated conditions and (b) samples after 20 days in accelerated conditions along with tolerance range (solid lines show dielectric constant ($\varepsilon_r^{'}$) and dashed lines show electrical conductivity (σ))

Similarly, figure 2(a) and figure 2(b) present comparison of three sample sets of composition B, under accelerated conditions for 10 days and 20 days respectively, along with the 5% tolerance band.

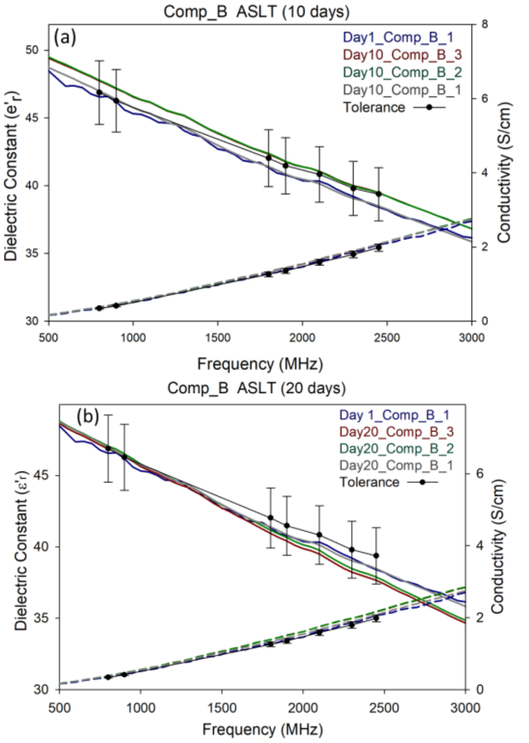

Figure 2. Comparison of dielectric properties of composition B at start of test (Comp_B_1 Dark blue) with (a) Samples after 10 days in accelerated conditions and (b) samples after 20 days in accelerated conditions along with tolerance range (solid lines show dielectric constant ($\varepsilon_r^{'}$) and dashed lines show electrical conductivity (σ))

It is evident from the figures that both the compositions show deviation in their dielectric properties due to exposure to accelerated conditions. However, this variation is quite small and well within the tolerance band for the duration of study.

After completion of this study, real time shelf-life study was started. One sample for each composition (namely Comp_A_4 & Comp_B_4) was kept under regulated condition from the start of study for duration of 18 months and dielectric properties were measured at specific intervals.

3.2 Real-world Shelf-life Study

The study of dielectric properties of both the compositions Comp_A & Comp_B, stored under regulated conditions (i.e. temperature between 18°C and 25°C (within ±1 °C) with RH between 40%-60%), has been shown in figure 3 and figure 4. The measurements are obtained at specified intervals i.e. at start of study (Dark blue) and at the end of 3rd month (dark red), 12th month (dark green), 15th month (dark cyan) and 18th month (dark grey).

The composition A (Comp_A shown in figure 3) has dielectric properties close match to IEEE specified values at 834 MHz & 900 MHz, which corresponds to 2G communication band. While, composition B (Comp_B, shown in figure 4) has dielectric properties close to specified values between 1450 MHz-250 MHz, which correspond to 3G, 4G & WiFi communication bands. The tolerance bands for the IEEE specified target values have also been shown in both the figures, to allow to determine the usable shelf-life of TEL compositions at different frequencies.

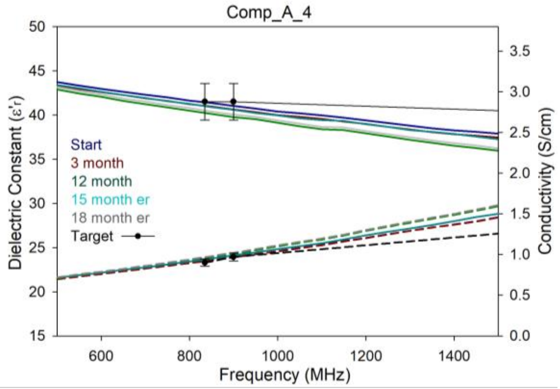

Figure 3. Study of dielectric properties of TEL composition A (Comp_A) for a period of 18 months along with IEEE specified target values at 835 MHz & 900 MHz (solid lines show dielectric constant ($\varepsilon_r^{'}$) and dashed lines show electrical conductivity (σ))

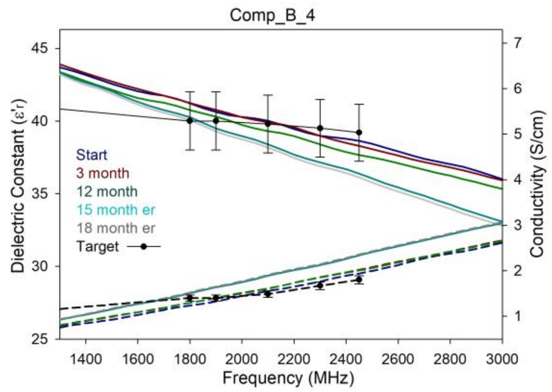

Figure 4. Study of dielectric properties of TEL composition B (Comp_B) for a period of 18 months along with IEEE specified target values between 1450MHz-2500 MHz (solid lines show dielectric constant ($\varepsilon_r^{'}$) and dashed lines show electrical conductivity (σ))

It can be clearly observed that the dielectric properties degrade during the duration of study. However, this degradation was slower at lower frequencies for both the compositions and faster at higher frequencies. It was also seen that composition A has degraded at much lower rate compared to composition B.

4. Conclusion

The accelerated and real-world shelf study of two Tissue Equivalent Liquid compositions was carried out. It was found that although accelerated conditions showed variation in dielectric properties of TELs, it was well within the 5% tolerance band. Hence, the shelf-life of TEL compositions could be established by real world stability study. It was found that one of the compositions (Comp_A containing DI water, DGBE and salt) degraded much slower than the other composition (Comp_B containing DI water, DGBE, Triton-X and sugar). The dielectric properties for composition A, remained well within the tolerance band for entire 18 months duration compared to 12 months duration for composition B.

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