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Differences in Rust in Hearing Aid Batteries across Four Manufacturers, Four Battery Sizes, and Five Durations of Exposure

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Abstract

Three hundred twenty zinc-air batteries representing four manufacturers (Energizer, Power One, Duracell, and Ray-O-Vac) and four cell sizes (10, 312, 13, and 675) were exposed in a salt spray fog apparatus for 2.5, 5.5, 24, 48, and 72 hours. At the conclusion of each exposure, the batteries were rated blindly for the presence of rust by four experienced audiologists using a four point rating scale. Results revealed significant differences in the rating of rust across the four manufacturers and duration of exposure. No statistically significant difference was found across cell size. Also, the correlation between raters was exceptionally high indicating that each audiologist rated the presence of rust for each battery in a very similar manner. Scanning electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDS), and Auger electron spectroscopy (AES) techniques were applied and provided answers for the observed differences in rust between the four manufacturers.

Key Words: Auger electron spectroscopy, corrosion, Duracell, Energizer, energy dispersive x-ray spectroscopy, hearing aid battery, Power One, Ray-O-Vac, rust, salt spray fog apparatus, scanning electron microscopy, zinc air

Abbreviations: AES = Auger electron spectroscopy; ASTM = American Society for Testing Materials; BTE = behind-the-ear; C = carbon; Cl = chlorine; Cr = chromium; D = depth; EDS = energy dispersive x-ray spectroscopy; ITE = in-the-ear; F = Fahrenheit; Fe = iron; H = height; KeV = kilo electron volts; N = nitrogen; Na = sodium; NaCl = sodium chloride; Ni = nickel; O = oxygen; PSI = pressure per square inch; RH = relative humidity; SEM = scanning electron microscopy; Si = silicon; W = width

Sumario

Trescientas veinte batería de aire-zinc, pertenecientes a cuatro fabricantes (Energizer, Power One, Duracell, and Ray-O-Vac) y cuatro tamaños de baterías (10, 312, 13, and 675) fueron expuesta a un aparato de neblina de atomización de sal durante 2.5, 5.5, 24, 48, y 72 horas. Al final de cada exposición, las baterías fueron evaluadas en forma ciega por cuatro audiólogos experimentados buscando la presencia de herrumbre, por medio de una escala de cuatro puntos. Los resultados revelaron diferencias significativas en la calificación del herrumbre en relación a los cuatro fabricantes y la duración de la exposición. No se encontraron diferencias estadísticamente significativas en cuanto al tamaño de las baterías. También, la correlación entre evaluadores fue

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excepcionalmente alta, indicando que cada audiólogo valoró la presencia de herrumbre para cada batería en una forma muy similar. Se aplicaron técnicas de microscopía electrónica de escaneo (SEM), de espectroscopía de rayos x de energía dispersante (EDS) y de espectroscopía electrónica Auger (AES), y éstas aportaron respuestas para las diferencias observadas en el herrumbre entre los cuatro fabricantes.

Palabras Clave: Espectroscopía electrónica Auger, corrosión, Duracell, Energizer, espectroscopía de rayos x de energía dispersiva, batería de auxiliar auditivo, Power One, Ray-O-Vac, herrumbre, aparato de neblina por atomización de sal, microscopía electrónica de escaneo, zinc-aire

Abreviaturas: AES = espectroscopía electrónica Auger; ASTM = Sociedad Americana de Evaluación de Materiales; BTE = retroauricular; C = carbón; Cl = cloruro; Cr = cromo; D = profundidad; EDS = espectroscopía de rayos x de energía dispersante; F = Fahrenheit; ITE = intra-auricular; Fe = hierro; H = altura; KeV = voltio kilo electrónicos; N = nitrógeno; Na = sodio; NaCl = cloruro de sodio; Ni = níquel; O = oxígeno; PSI = presión por pulgada cuadrada; RH = humedad relativa; SEM = microscopía electrónica de escaneo; Si = silicón; W = ancho

The first three authors are experienced audiologists who noticed differences across battery manufacturers in the presence of rust in the battery compartment. An extensive search using the major search engines and databases (SCOPUS, Web of Science, PubMed, Google Scholar, WorldCat, ArticleFirst, CINAHL, ComDisDome, Applied Science, and INSPEC) and key words such as “sweat,” “perspiration,” “corrosion,” “corrosive,” “rust,” “hyperhidrosis,” “battery,” “batteries,” “hearing aid batteries,” “metals,” “high sweat production,” and “low sweat production” could not locate any peer-reviewed studies investigating differences in rust production across battery manufacturers. It was these observations (i.e., presence of a problem and little research available to provide an answer) that served as the catalyst for this study.

A zinc-air battery is essentially a transducer that converts one form of energy (chemical) to another form of energy (electrical). The magnitude of the converted energy is related to the size of the anode (negative) and cathode (positive) material, zinc metal, an electrode with a small amount of internal stored oxygen and access to additional oxygen from the air surrounding the battery. When the small amount of internal oxygen is used, the battery looks for oxygen outside the battery. This is why air holes are in the bottom (positive side) of the zinc-air battery can. These holes allow air to access the cell to replenish the oxygen supply which mixes with the zinc metal and creates the electrical

discharge (McKenzie, 2003).

One problem with hearing aid batteries is their exposure to the environment, making batteries susceptible to the chemicals contained within sweat (predominantly sodium chloride [NaCl], but also potassium, urea, creatine, creatinine, lactate, and phosphate). The “normal” concentration of NaCl in sweat at rest is approximately 1.8 grams/liter, where a liter is a little more than a quart (Lind, 1972). This amount of NaCl converts to a concentration of approximately 0.2% by weight of NaCl in sweat. There is, however, significant inter and intra subject variation in the concentration of NaCl in sweat. This variation can depend upon the intensity and duration of exercise, sweat rate, sodium loss ratio, state of physical fitness, gender, age, diet, type and amount of clothing worn, temperature, relative humidity, acclimatization, level of hydration, and diseases such a pancreatic cystic fibrosis which has a significantly higher concentration of NaCl in sweat. For example, sweat can contain 0.8 to 2.0 grams/L (800 to 2000 milligrams) of NaCl in a heat-acclimatized individual and 3.0 to 4.0 (approximately 0.3% to 0.4% by weight) grams/L (or more) in a non-acclimatized individual. Added to this is that sweat rates can vary by 1 to 3 liters per hour with subsequent greater loss of NaCl. ISO 3160/2 (1982) is an international standard for the composition of “artificial sweat.” This standard calls for the amount of NaCl in sweat to be 20g/liter. This would convert to a concentration of approximately 2% of NaCl by weight per liter. The

artificial sweat standardized in ISO 3160/2 (1982) is used to assess the impact of sweat on products (jewelry; cosmetically treated hair) that come into direct and prolonged contact with the skin (Randin, 1988; Skopp et al, 1997). Thus, it can be stated that the concentration of NaCl in sweat can vary from as little as 0.2% to as great as 2% (Verde et al., 1982; Randin, 1988; Skopp et al., 1997). In addition, Buckley and Lewis (1960) reported that a concentration of as little as 0.5% to 0.85% of NaCl in sweat can begin to corrode mild steel.

Unfortunately, NaCl in sweat serves as an electrolyte promoting ionic conduction resulting in rust (Carpenter, 2003). There are differences in the material used to manufacture the cathode can (positive side) of a zinc-air battery as well as the manner in which the battery is produced. These differences can vary considerably between manufacturer and may explain why some batteries are more resistant to rust than others. For example, some manufacturers use stainless steel that is less prone to corrosion, while others use steel and then plate (i.e., cover) the steel using nickel (i.e., nickel plated) in a process called *post-plating*. Other manufacturers pre-plate the steel using nickel and then make the cathode can using a process called *pre-plating*. There are also differences in plating methods. For example, some manufacturers use an electro-less method that does not use electricity as part of the process. This method tends to provide a relatively thin plate to cover the steel and it is not very ductile (i.e., can crack). Others use an electrolytic method where electrons are supplied from a DC source. This method tends to yield a thicker plate to cover the steel that is more ductile (i.e., less prone to crack). Finally, most manufacturers use a tri-layer anode (negative side) cup where nickel is on the outside, stainless steel in the middle and copper on the inside that interfaces with the zinc anode mix on the inside of the battery. This material, in combination with the nickel covering the steel, can provide good resistance to rust.

The process of forming the cathode may damage the nickel plating leaving exposed cracks for moisture to come into contact with the steel below the nickel covering. These cracks can also serve to expose the middle steel layer of the anode cup causing a greater probability of producing rust. Finally, the edge where the can is punched from the strip

stock of steel is typically unplated and all these spots can rust. Any crack present when a manufacturer closes the cathode can during production is a potential spot for corrosion. Use of the thin plating technique (i.e., electro-less) might not be sufficient to cover these surface imperfections, whereas the electrolytic method will provide greater protection.

The impact of battery rust on hearing aid performance is probably minimal at first until the buildup, occurring over time, corrodes the battery contacts and/or wires within the battery compartment. This process can take a considerable amount of time that can eventually lead to intermittency or a "dead" hearing aid. Although not studied in the present investigation, the presence of rust could also act as a "stand-off," pushing the battery away from the contacts, resulting in loss of performance. This would be somewhat analogous to a flashlight sometimes providing a brighter light when the battery cap is screwed on tighter. In a hearing aid, better battery contact could provide better clarity and longer battery life (Phil Cooper, pers. comm., 2007). Finally, the presence of rust in the battery compartment, even if not impacting hearing aid performance, is simply unacceptable and unsightly just as the presence of dirt on any consumer product is unacceptable even though its presence has little or no impact on performance. For some patients, rust in the battery compartment is a source of embarrassment because they feel the audiologist may question how well they care for their aids as well as being reflective of their overall personal hygiene.

The presence of battery rust can be of significant clinical importance for professionals and patients dealing with issues related to amplification. Professionals and consumers would be interested to know if differences in rust are present as duration of exposure increases, as well as between battery manufacturers and battery sizes. This information might be beneficial when professionals counsel their patients on the use and care of batteries as well as possibly "steering" patients to one manufacturer or another because of differences in the development of rust. In addition, this information might be of interest to patients who reside or visit regions of the world where humidity is higher because greater humidity typically results in greater sweat. This would be especially true for

patients who have a tendency to sweat excessively. For example, patients, or parents of patients, involved in athletics would be interested because research has shown that the sodium concentration in sweat during exercise increases significantly relative to sodium concentration in sweat when at rest (Robinson et al, 1956; Kozłowski and Saltin, 1964; Shirreffs et al, 2005). Another example of patients who sweat more than usual is a sub-group of workers within industries making metal products that are known as “rusters” (Buckley and Lewis, 1960; Burton et al., 1976; Jensen, 1979; Jensen and Nielsen, 1979). In these workers, their NaCl concentration is so high that they can cause metal to rust in a short time by simply touching any metal component used to make the product. For these workers, a screening test has been developed and procedures made available to reduce the concentration of NaCl in their sweat while working.

In an attempt to determine how widespread the problem of rust in the battery compartment may be, the primary author contacted eight major hearing aid manufacturers to determine what percent of annual hearing aid repairs were related *specifically* to rust in the battery compartment. While one manufacturer reported such data was not available because this problem is combined into the general category of “moisture related problems,” another manufacturer reported this problem was present in less than 1% of repairs. Still another manufacturer stated it was present in about 3-5% of behind-the ear (BTE) aids and almost never in in-the-ear (ITE) aids. Another manufacturer reported battery rust was present in about 35% of behind-the-ear (BTE) and 5% of in-the-ear (ITE) repairs. Yet another manufacturer stated that battery contacts are replaced on almost all devices that are forwarded for repair because “any unit that has been in use for any length of time will have at least a little discoloration and the manufacturer wants to ship the device back to the patient looking new.” In addition, some manufacturers provide batteries when new hearing aids are shipped. Recently, at least one manufacturer changed battery vendors because use of a previous vendor resulted in battery rust that resulted in excessive repairs and a poorer than desired image upon the manufacturer. Viewing the sum of the information derived from the manufac-

turers, as well as the other factors cited above, it is the opinion of the authors that battery rust should not be dismissed as a trite or insignificant matter.

With these thoughts in mind, the authors engaged in a study to investigate possible differences in rust across four battery sizes used in hearing aids (10, 312, 13 and 675) and across four major battery manufacturers (Energizer, Power One, Duracell, and Ray-O-Vac). To assess possible differences in rust, a large sample of the four battery sizes from the four manufacturers were exposed to salt water in a salt spray fog apparatus for durations of 2.5, 5.5, 24, 48, and 72 hours. For the experimental conditions (four manufacturers, four battery sizes and five durations of exposure), four experienced audiologists independently rated the presence of rust using a four point interval scale. At the conclusion of the study, the investigators hoped to answer the following major questions:

1. Were significant differences present in rust as the duration of exposure increased for the intervals investigated in this study?
2. Were significant differences present in rust between the four battery manufacturers investigated in this study?
3. Were significant differences present in rust for the four battery sizes investigated in this study?
4. What was the relationship in rating the presence or absence of rust between the four raters across the four test runs?
5. If significant differences are present then what may account for those differences?

METHODOLOGY

Battery Purchase

Ninety-nine individual packages of batteries were purchased by an independent firm from nine cities (Chicago, Minneapolis, Philadelphia, St. Louis, Houston, Seattle, Los Angeles, Miami, and Burr Ridge) and eleven sources (Osco, Meijer, Walmart, CVS, Menards, Target, Albertson's, Longs, Walgreens, Micropower Battery Company, and HITEC group). These batteries were representative of the four sizes of zinc-air batteries (10, 312, 13, and 675) and the four manufacturers (Energizer, Power One, Ray-O-Vac, and Duracell) used in the current study. The expiration date on each package ranged from January 2008 to December

2009. Upon arrival, each package was provided an identification number (ID) (1-99) and an Excel spreadsheet was created documenting the city, name of store, battery size, manufacturer, number of batteries within the package (four to 16), emboss-tracking number, and expiration date.

Identification of Batteries and Assigning Batteries for Testing

The spreadsheet containing the ID numbers for the 99 packages of batteries (representing 1570 total purchased batteries from the four manufacturers and four cell sizes for sixteen possible combinations) were provided to a statistician. The statistician entered the 99 ID numbers into a statistical software package that generated twenty random orders of the sixteen possible combinations of the four manufacturers and four cell sizes. This ensured that each of the sixteen combinations was tested 20 times. The orders were generated using a Latin Square methodology to ensure that no particular manufacturer or battery size appeared consistently in a particular ordinal position of a sequence of 16. These orders were then chained together to generate the 320 (ID numbers of 1-99) positions across eight cards. Figure 1 provides the order for placement of each cell from the batches 1-99 on the eight acrylic cards where each card contained 40 batteries across eight columns and five rows for a total of 320 cells. For example, in Figure 1, the numbers in the cells placed in the first row for Card #1 corresponded to the battery pack ID 93, 94, 60, 84, 34, 91, 20, and 67. In this example, these corresponded to Power One #675, Power One #10, Duracell #10, Duracell #312, Energizer #675, Power One 312, Energizer #10, and Duracell #312, respectively.

The authors carefully followed the outline for battery placement illustrated in Figure 1 to place the 320 batteries onto the eight acrylic cards. First, five evenly spaced rows of double sided tape were placed on each of the eight acrylic cards. When removing the batteries from their packs and placing them on the cards, the positive side was placed on the tape so that the negative side was facing out. Also, when removing the batteries from the packs and placing the batteries onto the cards, the authors used rubber gloves so oil or debris from our fingers did not contaminate the batteries. Each card was then

placed into a plastic zip-lock bag after the batteries were placed on the card. The card was not removed from the zip-lock bag until placed into the salt spray fog apparatus.

Rating Rust

Rating Scale

The following table is illustrative of the rating scheme used by four experienced audiologists to independently rate the magnitude of rust on each battery once the batteries were removed from the salt spray fog apparatus.

Rating	Corrosion
0 = No rust observed at via the naked eye or 10x magnification.	None
1 = Rust observed only at 10x magnification.	Light
2 = Rust <i>noticeable</i> via the naked eye.	Moderate
3 = Rust <i>easily observed</i> via the naked eye.	Heavy

Thus, a rating of “0” (None) meant that no rust was observed via the naked eye or when observed under the microscope using 10x magnification. A rating of “1” (Light) meant that light rust was not observed via the naked eye, but was observed under the microscope using 10x magnification. A rating of “2” (Moderate) meant that rust was *noticeable* via the naked eye, while a rating of “3” (Heavy) meant that rust was *easily observed* via the naked eye.

The photographs in Figure 2 were placed on the wall to the side of the microscope used for rating as a reminder of the rating scale. Also, the rating scale was repeated at the bottom of each scoring sheet that the audiologists used to rate each battery. Each test run contained two cards (80 batteries). Thus, two cards were placed in the salt spray fog apparatus each week. Each of the two cards was removed from the salt spray fog apparatus at 2.5, 5.5, 24, 48, and 72 hours of exposure. The total project ran for four weeks. Cards #1 and #2 were placed in the apparatus in the first week; Cards #3 and #4 were placed in the apparatus the second week; Cards #5 and #6 were placed in the apparatus the third week; and Cards #7 and #8 were placed in the apparatus the fourth week.

Card	1							
	93	94	60	84	34	91	20	67
	37	71	82	92	17a	19	16a	3
	5	12	18	97	67	90	27	36
	1	55	95	24	86	92	40	79
	37	72	95	4	32	62	28	92
Card	2							
	54	97	66	60	90	64	17	1
	79	33	92	32	48	27	90	29
	97	30	26	95	85	34	77	37
	2	14	93	29	95	46	30	18
	85	50	77	1	43	90	96	78
Card	3							
	84	24	22	75	2	48	44	47
	65	90	50	58	96	93	13	95
	91	71	35	16a	7	14	93	90
	43	11	46	17a	63	62	61	96
	94	18	61	15	92	65	35	93
Card	4							
	76	86	72	34	95	22	47	85
	16	91	3	10	51	69	13	52
	30	96	94	45	46	58	97	65
	92	93	9	49	38	43	95	19
	94	42	45	26	60	20	28	8
Card	5							
	64	55	90	92	63	59	7	15
	95	22	6	57	93	41	80	25
	29	82	55	62	86	61	92	91
	51	47	49	93	57	38	90	71
	25	96	39	94	84	11	36	81
Card	6							
	64	91	33	6	53	83	52	97
	6	17	20	91	73	97	32	44
	28	83	92	66	4	72	87	94
	63	83	67	74	90	96	10	8
	57	35	93	5	80	27	91	81
Card	7							
	75	13	53	88	93	89	73	76
	92	9	7	94	41	95	74	68
	69	96	97	2	94	36	70	89
	91	40	11	41	68	31	38	75
	97	95	31	90	54	80	96	52
Card	8							
	70	89	16	81	40	39	12	33
	91	45	26	59	8	23	58	88
	21	68	56	96	48	94	97	31
	23	88	96	97	73	76	94	87
	5	91	44	78	42	12	56	21

Figure 1. The battery identification (ID) numbers used to place the 320 batteries on the eight acrylic cards.

Raters

Four experienced audiologists with three, five, 23 or 26 years of experience served as the raters. Each audiologist *independently* observed and rated each of the 320 batteries at 2.5, 5.5, 24, 48, and 72 hours. Using a single blinded procedure, no rater had any knowledge of the battery manufacturer that was being rated. In addition, no rater was aware of the rating reported by any of the other raters.

Salt Spray Fog Apparatus

Figure 3 illustrates and identifies the major components of the MX-9208 Salt Spray Fog apparatus used in this study. Not shown is the Panther Compact 106 compressor (115V/60 Hz) used to generate the pressure for the line and water tower tank. On the compressor were two gauges (line and tank pressure gauges). The line pressure gauge was adjusted to 25 psi (red scale) or 1.75 μ bar (black scale). A regulator was used and the gauge was set at 16.0 psi (+/- 0.5 psi). The typical compressor cycle for reference purposes was 17 seconds regeneration time (compressor on) with a 1 minute 20 second dwell time (compressor off).

The MX-9208 Salt Spray Fog apparatus is a double-walled chamber used for corrosive atmospheric tests and was operated during the course of the study as described in the standard by the American Society for Testing

Figure 2. Four categories used to rate battery rust.

Figure 3. MX-9208 Salt Spray Fog apparatus and the major components.

Materials (ASTM) B117 (2003). This apparatus allows for the temperature between the two walls to provide warm air circulation, by blowers and a heater located at the base, between the inner and outer walls. The thermostat within the test area of the apparatus is factory set to control the temperature to 95° F (+/- 2° F). When the chamber is operated in accordance with the ASTM B117 practice, the relative humidity (RH) within the chamber is between 95 and 98%. According to Section 6 of ASTM B117, RH is not a measure that is recorded or necessary when the apparatus is properly maintained as indicated by the psi, temperature, and accumulation measures.

The saturation water tower was adjusted for a temperature of 112° F (+/- 2° F) and a pressure reading of 12 psi (+/- 0.5 psi). Prior to each weekly run, the water level in the water tower was filled with *distilled water* to 2" below the cover.

For each of the four runs, two acrylic cards with 40 batteries each (total of 80 batteries) were suspended on two lucite rods within the apparatus (negative side of the battery facing down toward the inside of the apparatus). This prevented condensation of the salt spray fog directly onto the battery which could short the anode and cathode thus promoting galvanic corrosion. Clean, compressed air from the compressor was connected to an air valve at the rear of the apparatus. This air at 12-15 psi was bubbled through a saturation tower containing distilled water and was heated to 112° F (+/- 2° F). The dry air then becomes humidified and enters the apparatus. The saturated air passes over a siphon tube that is immersed in a salt water reservoir. By means of suction, the salt solution is

mixed with the air and atomized through the nozzle creating a "fog." The preparation of the salt solution consists of sodium chloride containing no more than 0.1% of sodium iodide and no more than 0.3% of total impurities. The 5% salt solution was purchased in a 50 gallon drum from National Exposure Testing Inc (NET) and is certified by NET to be in compliance with ASTM B117 (2003) specifications. The solution was manufactured using Reagent Grade Size IV deionized water. During testing, the salt concentration was checked per ASTM B117-03 at 24, 48 and 72 hours of exposure using a digital specific gravity hydrometer and measurements were always within the specified range of 1.0255 and 1.0400 g/mL with the water temperature measured between 76-78° F. In addition, two clean fog collectors were strategically placed within chamber so no drops of solution were collected from the batteries. The atomization and quantity of the fog was such that the collection rate was 1.0 to 2.0 mL of solution per hour within each collector based on an average run of 16 hours. The inner working volume of the apparatus is maintained at 95° F by means of hot air being circulated in the cavity around the inner apparatus. There is a water trough along the top of the apparatus where the cover comes into contact with the top of the apparatus. This trough is completely filled with distilled water to create a seal so that the salt spray fog does not enter the room where the salt spray fog apparatus resides.

The MX-9208 has a salt solution capacity of 11 gallons (41 liters). The saturation tower has a capacity of 2 gallons (10 liters). Each can operate up to 72 continuous hours. The internal working dimension of the salt spray fog apparatus is 24" (H) x 24" (W) x 24" (D). The external dimensions are 37" (H) x 48" (W) x 34" (D).

The MX-9208 and the procedures used in this study adhere to the ASTM B117-03 *Standard Practice for Operating Salt Spray (Fog) Apparatus* (ASTM, 2003).

Scanning Electron Microscopy (SEM)/Energy Dispersive X-ray Spectroscopy (EDS)

SEM images of batteries were acquired using a JEOL Model 6300F field-emission instrument. An Oxford Model 6731 EDS attachment provided compositional analysis

Figure 4. Low magnification (10x) scanning electron microscopy (SEM) of a hearing aid battery identifying the cathode, cathode radius, and anode.

of the areas of interest. The depth of analysis of EDS is on the order of a few microns. A fresh unused sample of each of the four brands of batteries was removed from its packaging and mounted directly onto a standard 12 mm sample stub and introduced into the high-vacuum chamber. The SEM and EDS analyses were performed using a 10 KeV (kilo electron volts) primary electron beam. Since the salt spray fog test showed corrosion primarily in the cathode radius (Figure 4) this is the area that was studied.

Auger Electron Spectroscopy (AES)

Auger electron spectroscopy (AES) is an ultra-high vacuum technique involving irradiation of solid surfaces with a finely focused beam of electrons (primary electrons). As the

primary electrons strike the surface atoms, Auger electrons are emitted from the top ~5 nm (nanometers) of the sample. The discrete energies of the Auger electrons are measured and are specific to the element from which it came. The Auger data were acquired using a Physical Electronics Model 680 Field-Emission Auger instrument. The same batteries as those analyzed with SEM and EDS were used for the Auger studies. The primary electron beam was operated at 10 KeV to obtain both secondary electron images and Auger data. The depth of analysis for the Auger technique is on the order of 5 nm. To remove atmospheric contamination, a slight inert gas sputter cleaning of the samples was performed prior to Auger analysis. This was accomplished using a 2 KeV argon ion etch within the Auger system.

RESULTS

The ratings for rust for the three independent variables of *duration of exposure* (five treatment levels of 2.5, 5.5, 24, 48, and 72 hours), *manufacturer* (four treatment levels of Energizer, Power One, Duracell, and Ray-O-Vac), and *battery size* (four treatment levels of 10, 312, 13, and 675) were analyzed using a three-factor repeated randomized analysis of variance (ANOVA) and post-hoc analysis to identify any significant differences. The magnitude of agreement between the four raters across the four test runs was analyzed using Pearson product correlation.

Figures 5 to 7 summarize the major findings from this study achieving statistical sig-

Figure 5. Cumulative percent of rating (0-3) as a function of duration of exposure (2.5, 5.5, 24, 48, and 72 hours).

Figure 6. Mean rating (0-3) as a function of manufacturer (Energizer, Power One, Duracell, and Ray-O-Vac).

nificance. Figure 5 reports the cumulative percent of ratings (0 to 3) as a function of duration of exposure (2.5, 5.5, 24, 48, and 72 hours) collapsed over manufacturer and battery size. Figure 6 describes the mean rating for each of the four manufacturers collapsed over duration of exposure and battery size. Figure 7 reports the mean rating as a function of manufacturer and duration of exposure collapsed over battery size.

A repeated randomized block ANOVA (Kirk, 1982) was performed on the data appearing in Figures 5 to 7. The ANOVA revealed a significant main effect for time of exposure (Figure 5) ($F = 502.85$; $df = 1.925$; 1204 ; $p < 0.001$) collapsed over manufacturer and battery size. As expected, as the duration of exposure increased from 2.5 hours to 72 hours, there was a significant increase in the presence of rust (i.e., gradual decrease in rating of 0 and a gradual increase in the number of rating of 3). That is, 60% of the batteries had a rating of “0” after 2.5 hours of exposure and this rating decreased to approximately 18% by 72 hours of exposure, whereas less than 3% had a rating of “3” at 2.5 hours of exposure and this rating increased to approximately 52% at 72 hours of exposure. It is interesting to note that in spite of 72 hours of exposure, approximately 18% of the batteries still maintained a rating of “0.” Post-hoc analysis using the t-test revealed that the mean rating for each of the five durations was significantly different ($p < 0.0001$) than the mean rating for any other duration of exposure.

The ANOVA also revealed a significant main effect for manufacturer (Figure 6) ($F = 145.37$; $df = 3$, 301 ; $p < 0.001$). Post-hoc analysis using the t-test ($p < 0.0001$) revealed that the mean ratings for Energizer and

Figure 7. Mean rating (0-3) as a function of manufacturer and duration of exposure.

Power One were significantly better (lower) than the mean ratings for Duracell and Ray-O-Vac (higher). In addition, there were no significant differences in the mean rating between Energizer and Power One or between Duracell and Ray-O-Vac.

The ANOVA revealed a significant time of exposure by manufacturer interaction (Figure 7) ($F = 13.87$; $df = 5.775$, 1204 ; $p < 0.001$). The odd fractional degrees of freedom are due to the use of Greenhouse-Geisser corrections for violation of sphericity. Figure 7 reports that as duration of exposure increased from 2.5 to 72 hours, the mean ratings for the four manufacturers became poorer (i.e., higher value). Post-hoc analysis using the t-test revealed that the mean rating for each of the five durations was significantly better ($p < 0.0001$) for Energizer and Power One than between Duracell and Ray-O-Vac. Also, for each duration the mean rating was not significantly different between Energizer and Power One or between Duracell and Ray-O-Vac. The mean rating, however, for each duration of exposure for Energizer and Power One was significantly better (lower) than the mean rating for Duracell and Ray-O-Vac (higher). Finally, the main effect of battery size; the two-factor interactions of battery size by manufacturer, and battery size by time of exposure; and the three-factor interaction of battery size by manufacturer by time of exposure were found not to be significant.

Surface Characterization

From the data reported above it is clear that different battery manufacturers produced batteries that varied significantly in terms of resistance to rust. To determine why

Figure 8. SEM image (100x) of Duracell battery showing fissures in nickel plating along the cathode radius region.

this was true, morphological (SEM) and compositional (EDS and AES) testing of the battery surface was undertaken. To minimize out-gassing of the batteries within the vacuum test chambers, the #10 battery of each manufacturer was selected for testing because the salt spray fog tests clearly reported that battery size was not a factor in the degree of rust. As reported earlier, Figure 4 shows a low magnification (10X) secondary electron image of one of the Duracell batteries. Since visually much of the rust appeared

Figure 9. Increased magnification (2000x) of SEM of a Duracell battery showing fissures along cathode radius. Spectrum 1 points to a fissure, while Spectrum 2 points to an area of nickel plating.

to be located on the upper curved surface of the cathode (cathode radius), SEM images were obtained from this region on all four batteries. Figure 8 shows a higher magnification (100X) of the cathode radius on the Duracell battery. Several cracks or fissures are readily observed in this curved section of the cathode. An even higher magnification image (2000X) of some of the representative fissures on the Duracell battery is shown in Figure 9. In this figure, "Spectrum 1" high-

Figure 10. Energy dispersive x-ray spectroscopy (EDS) spectra of a Duracell battery for measurements made from inside the fissure (upper-spectrum 1) and on the nickel plating (lower- spectrum 2). Notice the peaks in Fe (iron), along with some Ni (nickel), found in the fissure (upper) and the peaks of predominantly nickel, along with some iron, found in the plating (lower). In EDS analysis, the ordinate is x-ray intensity and the abscissa is x-ray energy.

Figure 11. SEM (left) at 2000x magnification and Auger spectra (right) of a Ray-O-Vac battery where “1” is inside the fissure and “2” is on the nickel plating. Notice the large amounts of Fe and Ni found inside the fissure (right-upper graph) and the high amounts of Ni found in the plating (right-lower graph). In Auger analysis, the ordinate is electron intensity and the abscissa is electron energy.

lights a fissure, while “Spectrum 2” highlights a region of nickel plating. EDS analysis (in Figure 10) of an area (800 μm x 600 μm) outside the fissure (lower curve of spectrum 2) shows a little Fe (iron), C (carbon), and P (phosphorus), but predominantly spikes of Ni (nickel). EDS analysis within a fissure (upper curve of spectrum 1) shows a little Ni and P, but predominantly spikes of Fe. Since the depth of analysis of EDS is on the order of a few micrometers, EDS cannot rule out the possibility that a thin layer of protective nickel is still present on top of the iron within these fissures, it is conceivable that the iron is totally exposed within the cracks and therefore prone to corrosion. SEM (left image in Figure 11) and EDS (right in Figure 11) analysis of the Ray-O-Vac battery produced

results very similar to the Duracell battery with deep narrow fissures observed along the cathode radius. EDS analysis of an area outside the fissure shows O (oxygen), but predominantly spikes of Ni. EDS analysis inside the fissure show O and Ni, but predominantly spikes of Fe. The Power One battery also showed cracks in the nickel plating (Figures 12 and 13); however, the cracks were typically wider and not as deep as those found on the Duracell and Ray-O-Vac batteries. Substantially more iron was again found inside the cracks than outside the cracks using EDS. The Energizer battery looked quite different than the other batteries in that the SEM images showed no signs of cracking in the outer nickel plating and very little iron was detected in the EDS spectra (Figure 14).

Figure 12. SEM (10x magnification) of Power One battery showing fissures in nickel plating along the cathode radius. Spectrum 1 points to the region of the nickel plating, while Spectrum 2 points to an area of a fissure.

Figure 13. Increased magnification (2000x) of SEM of Power One battery showing fissures along cathode radius and noting the Fe base material exposed in the fissure.

Figure 14. Increased magnification (2000x) of SEM of Energizer battery showing no fissures along cathode radius.

The hypothesis that cracking of the protective nickel plating of the batteries led to exposure of the underlying corrosion-prone iron fit the experimental salt spray fog rust results for the Duracell, Ray-O-Vac, and Energizer batteries. However, if cracking of the nickel plating is the problem, then the Power One batteries should also have performed poorly in the rust studies since cracks were also observed in the nickel plating on this battery. At this point Auger electron spectroscopy (AES) was chosen to study the compositions of the cracked regions of the batteries in a much more surface sensitive manner.

To determine if iron was present at the very surface of the batteries, AES were acquired from multiple areas, inside and outside of cracks, on each of the batteries. Iron was readily found within the cracks on the Duracell and Ray-O-Vac batteries, but was not readily observed on the Energizer or Power One batteries. Auger electron maps for iron were obtained on each of the samples and show the lateral distribution of iron present in the field of view (800 μm x 600 μm). AES shows that the presence of iron (i.e., “white spots”) follows the topography of the cracks on the Duracell (Figure 15) and Ray-O-Vac (Figure 16) batteries. On the Power One battery several small localized spots of iron were detected (Figure 17); however, the spots are much more random in location and less in number than the Duracell or Ray-O-Vac batteries. For Energizer, only a few very small specs of iron were detected on the surface (Figure 18).

Correlation between the Four Raters and Five Test Runs

Tables 1-4 report the Pearson correlation coefficients between the four raters for the four test runs. As can be seen, the correlations are remarkably high. This finding indicates that the same conclusion regarding rust would have been drawn regardless of which audiologist made the rating. A couple

Figure 15. SEM (upper) and corresponding Auger electron spectroscopy (AES) spectra (lower) iron map on a Duracell battery. The white “specks” in the AES spectra iron map reflect “spots” of iron detected using this procedure.

Figure 16. SEM (upper) and corresponding Auger electron spectroscopy (AES) spectra (lower) iron map on a Ray-O-Vac battery. The white “specks” in the AES spectra iron map reflect “spots” of iron detected using this procedure.

Figure 17. SEM (upper) and corresponding Auger electron spectroscopy (AES) spectra (lower) iron map on a Power One battery. The relatively few white “specks” in the AES spectra iron map reflect “spots” of iron detected using this procedure.

Figure 18. SEM (upper) and corresponding Auger electron spectroscopy (AES) spectra (lower) iron map on an Energizer battery. Notice only a very few white “specks” in the AES spectra iron map reflect “spots” of iron detected using this procedure.

Table 1. Pearson pair-wise correlations between Raters 1 to 4 for the first test of battery exposure.

Rater	1	2	3	4
1	1.00	0.82	0.75	0.55
2		1.00	0.80	0.46
3			1.00	0.55
4				1.00

Table 2. Pearson pair-wise correlations between Raters 1 to 4 for the second test of battery exposure.

Rater	1	2	3	4
1	1.00	0.87	0.83	0.76
2		1.00	0.86	0.78
3			1.00	0.79
4				1.00

Table 3. Pearson pair-wise correlations between Raters 1 to 4 for the third test of battery exposure.

Rater	1	2	3	4
1	1.00	0.88	0.88	0.82
2		1.00	0.87	0.85
3			1.00	0.82
4				1.00

Table 3. Pearson pair-wise correlations between Raters 1 to 4 for the fourth test of battery exposure.

Rater	1	2	3	4
1	1.00	0.92	0.91	0.88
2		1.00	0.91	0.88
3			1.00	0.88
4				1.00

of observations are worth noting. First, it can be seen that the correlation between the Raters 1-3 after Test 1 was rather high (.75 to .82), but poorer between Raters 1-3 and Rater 4 (.46 to .55). It became clear to the first author after reviewing the data from the first run that because of the wide discrepancy in the ratings reported by Rater 4 and Raters 1-3, (i.e., Rater 4 reported rust two to three intervals poorer than Raters 1-3) that perhaps it would be prudent to re-instruct Rater 4. As can be seen in Tables 2-4, the correlation between Rater 4 and Raters 1-3 improved considerably for Tests 2-4. The second major observation is the general trend of the correlations gradually improving from Tests 1-4 for all four Raters. That is, inter-Rater consistency improved with greater experience. Finally, Table 5 reports the Pearson pair-wise correlation after averaging the Raters 1-4 for Tests 1-4. This illustrates the high internal consistency of the four-judge composite rating for each time period.

Table 5. Pearson pair-wise correlation after averaging the four raters for each Test Run. This illustrates the internal consistency of the four-judge composite rating for each time period.

Test 1	0.88
Test 2	0.95
Test 3	0.96
Test 4	0.97

Discussion

From the author’s perspective, the major finding of this study was the presence of significant differences between Energizer and Power One in revealing less rust than Duracell or Ray-O-Vac under the same test conditions used in this study. One could argue, however, that the durations of exposure in this study may be far greater than usual when patients wear hearing aids during typical daily use. That is, users typically

Figure 19. Cumulative percent of rating (0-3) as a function of manufacturer for 2.5 hours of exposure.

do not wear hearing aids continuously for 24, 48, or 72 hours. Therefore, some might suggest that the results of this study may have little external validity to actual use in the “real” world. The authors, however, would argue that it is reasonable to inspect the data at 2.5 and 5.5 hours because these durations would not be unreasonable for many users in daily listening situations (running, biking, hiking, golfing, etc), those who sweat profusely and/or use the hearing aids in an environment with relatively high percent of humidity. Figures 19 and 20 report the cumulative percent of ratings for the four manufacturers at 2.5 (Figure 19) and 5.5 hours (Figure 20) of exposure. As can be seen in Figure 19, even for a relatively short duration of 2.5 hours, a “0” rating was achieved 88% and 81% of the time for Energizer and

Power One, respectively, while a rating of “0” was achieved 32% and 39% respectively for Duracell and Ray-O-Vac. If one were to expand the “acceptable criteria” to a rating of “1,” then the rate of acceptable performance would expand to 99% and 96% respectively for Energizer and Power One, while the rate of acceptable performance would expand to 76% and 79%, respectively for Duracell and Ray-O-Vac. If one were to view the data for 5.5 hours of exposure (Figure 20), a “0” rating was achieved 91% and 96% of the time for Energizer and Power One, respectively, while a rating of “0” was achieved 9% for both Duracell and Ray-O-Vac. Again, if one were expand the “acceptable criteria” to a rating of “1,” then the rate of acceptable performance would expand to 97% and 95% for Energizer and Power One, respectively, while the rate

Figure 20. Cumulative percent of rating (0-3) as a function of manufacturer for 5.5 hours of exposure.

of acceptable performance would expand to only 43% and 41% for Duracell and Ray-O-Vac, respectively.

One could also argue that the 5% NaCl solution used in this study is aggressive and that this amount of NaCl bears little resemblance to the concentration of NaCl in sweat in "real life" either under conditions of rest or exercise. Therefore, it could be argued once again that the results from this study have little external validity.

First, it must be remembered that the use of a 5% NaCl solution was required in order for the methodology to adhere to ASTM B117 (2003). Second, it needs to be emphasized that Sections 3.1 to 3.2.2 of ASTM B117 (2003) states,

this practice provides a controlled corrosive environment which has been utilized to produce relative corrosion resistance information for specimens of metals and coated metals exposed in a given test chamber. *Prediction of performance in natural environments has seldom been correlated with salt spray results when used as stand alone data.* Correlation and extrapolation of corrosion performance based on exposure to the test environment provided by this practice has not always been predictable. Correlation and extrapolation should be considered only in cases where appropriate corroborating long-term atmospheric exposures have been conducted.

Additional studies would be needed to determine if the results reported in this study, using the required 5% solution, would remain the same when using reduced concentrations of NaCl that are more compliant with the amount of NaCl reported in sweat at rest and during exercise. As mentioned in the introduction, the "normal" concentration of NaCl in sweat at rest is approximately 0.2% by weight. There is, however, significant inter and intra subject variation in the concentration of NaCl in sweat. This variation can depend upon intensity and duration of exercise, sweat rate, sodium loss ratio, state of physical fitness, gender, age, diet, type and amount of clothing worn, temperature, relative humidity, acclimatization, level of hydration, and diseases such as pancreatic cystic fibrosis which has a significantly higher concentration of NaCl in sweat. For example, as mentioned earlier, NaCl can be 0.2% to 0.4% by weight in a non-acclimatized individual. Added to this is that sweat rates can vary by 1 to 3 liters per hour with subsequent greater loss of NaCl. ISO 3160/2 (1982) is an inter-

national standard for the composition of "artificial sweat." This standard calls for 20g/liter of NaCl. This would convert to a concentration of approximately 2% of NaCl by weight per liter. Thus, it can be stated that the concentration of NaCl in sweat can vary from as little as 0.2% to as great as 2% (Verde et al., 1982; Randin, 1988; Skopp et al., 1997). Buckley and Lewis (1960) reported that a concentration of as little as 0.5% to 0.85% of NaCl can begin to corrode mild steel.

It is important to emphasize, however, that the batteries from the four manufacturers underwent the *same test conditions*. Thus, the authors would argue that the relative difference in rust buildup between battery manufacturers reported within the test apparatus does relate to real differences in the ability of the battery to resist rust/corrosion in the "real world." This, combined with the reports from manufacturers provided in the introduction, points to fact that (a) battery rust is a real problem (i.e., manufacturer reports of 1 to 35% of repairs are directly related to battery rust); and (b) differences in rust production across battery manufacturers is present (i.e., at least one manufacturer reported switching battery vendors due to reports of excessive rust from audiologists, patients and increased repairs). From the author's viewpoint, if a battery withstood the rigors of the procedure used in the present study and illustrated significantly less rust than another battery manufacturer under the same test conditions, it would seem reasonable to conclude the same battery would also yield relatively less rust in a less demanding environment than its competitor. That is, the same relationship of the absence or presence of rust found in the less demanding environment should be the same as it was in the more demanding environment. As mentioned earlier, the validity of this assumption needs to be tested in future research where the concentration of NaCl within the apparatus is at a level more typically reported under real world conditions.

The prevalence of battery rust requires at least two additional comments. First, excessive rust in the battery compartment is hardly ever the result of a single episode, but rather the cumulative effect of repeated use of the battery over time. That is, a relatively small amount of rust may be present after a single battery is used and then discarded

after 7-20 days of use. After repeated use, however, the accumulated rust will grow and build in the battery compartment causing, in extreme cases, poor or no contact with the battery contacts (i.e., intermittency or a dead hearing aid). Although not the focus of this study, up to that point, the presence of rust probably has minimal or no impact on the performance of the hearing aid. Second, the number of hearing aids sent to manufacturers for repairs due to battery compartment rust is probably smaller than reported because many audiologists implement at least two preventive steps. That is, these steps do not eliminate battery rust, but can serve to prevent the buildup from impacting hearing aid performance that would eventually require the need to send the aid for repair. First, as is the case in our facility, when patients are scheduled for an annual audiometric evaluation or services related to repair, one of the first actions taken by the audiologist is to check the battery compartment. If rust is observed, as it often is, the compartment is cleaned with a cotton applicator and contact cleaner. This finding leads to additional patient counseling and prevents the need to send the aid for repair. Second, as part of our practice, the audiologist provides an *electronic* desiccant device to every patient when hearing aids are dispensed. The audiologist counsels the patient to use the device each night and *keep the battery in the device overnight* to dry the hearing aid and battery. It is felt that if these practices (timely cleaning of the battery compartment, counseling, and use of an *electronic* desiccant device) were not implemented, the number of hearing aids sent for repair from our facility due to rust buildup would be significantly higher. Finally, assuming a lack of significant differences in battery performance and cost across manufacturers, it would seem reasonable that clinicians might look at possible differences in rust buildup as the decision related to battery purchase and recommendations to their patients is formulated.

FINAL IMPORTANT NOTE

Since the original submission of this manuscript, the first author presented the results at the American Auditory Society in March 2007. A short time later, the first author was contacted by a representative of Ray-O-Vac. The author was informed that

significant manufacturing changes had been made to the Ray-O-Vac batteries since the production of those used in the present study. The author was informed that Ray-O-Vac will introduce a new battery that uses post-plating electrolytic techniques in their ProLine Advanced battery. The author asked the representative to forward samples of the ProLine Advanced 10, 312, 13, and 675 batteries. These batteries were sent to EAG Laboratories where the last three authors completed SEM, EDS, and AES measures. The results of those measures found that the ProLine Advanced batteries revealed no cracks in the plating, and iron was not present as is reported for the original Ray-O-Vac batteries used in this study. Later, these new batteries were immersed in the salt spray fog chamber. The ratings were slightly higher but very similar to those reported for Energizer and Power One in this study. Therefore, the authors believe it is important that this change in manufacturing techniques in the Ray-O-Vac ProLine Advanced battery be brought to the attention of the reader.

CONCLUSIONS

The results of this study answered the following questions that were addressed in the introduction. That is:

1. Significant differences were present in rust as duration of exposure increased for the duration intervals used in this study. As duration of exposure increased there was an incremental increase in the presence of rust collapsed across manufacturer and battery size.
2. Significant differences were present in rust between the four battery manufacturers used in this study where there was significantly less rust and or corrosion for batteries manufactured by Energizer and Power One than for Ray-O-Vac and Duracell. In addition, within these two groups there were no significant differences in rust between Energizer and Power One or between Ray-O-Vac and Duracell.
3. Significant differences were not present in rust for the four battery sizes used in this study collapsed across duration of exposure and manufacturer.
4. Significant differences were present in rust for the two factor interaction of duration of exposure and manufacturer. That is, as duration of exposure increased mean ratings

for the four manufacturers decreased. The mean ratings, however, for each duration of exposure for Energizer and Power One were significantly better than the mean ratings for Duracell and Ray-O-Vac. In addition, for each duration of exposure, there were no significant differences between Energizer and Power One or between Duracell and Ray-O-Vac.

5. The correlation between the four raters across the four test runs was highly correlated and the correlation improved as the raters gained greater experience. That is, the correlation improved steadily from Test 1 to Test 4.

6. Surface analysis of the four batteries shows that cracks formed in the protective nickel plating on the Duracell and Ray-O-Vac batteries have left exposed iron present on the outermost surface. This iron is then prone to corrosion in the salt water test. Cracks were also discovered in the nickel plating on the Power One battery, but Auger analysis indicated that nickel was still present at the bottom of the cracks and iron was not exposed. The nickel plating on the Energizer battery showed no evidence of cracking.

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