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DUAL-CURING RESIN CEMENT WITH COLOR INDICATOR: CHANGE OF
COLOR BY CURING AND SOME PHYSICAL PROPERTIES

Syventävien opintojen kirjallinen työ
Kevätlukukausi 2019

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Hammaslääketieteen laitos
Kevätlukukausi 2019
Vastuhenkilö: Pekka Vallittu

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Hammaslääketieteen laitos, biomateriaalitiede
Maaliskuu 2019

Abstract

Objective. To investigate a color indicator containing dual curing resin composite luting cement and to plot the color change to the time of solidification of the cement. In addition some physical properties of the cement were studied after autopolymerization only, and with the light initiated polymerization.

Methods. Specimens were made of a dual-cure resin cement (Maxcem Elite™ Chroma, Kerr, Orange, CA, USA) and polymerized by autopolymerization only, or with light initiated polymerization. A spectrophotometer was used to quantify the color change of the cement as plotted with the curing time. The efficacy of the curing process was studied by measuring water sorption and the ultimate flexural properties of the cement.

Results. Flexural strength of cement after autopolymerization was 27.3 MPa (SD=5.1, n=8) and after light initiated polymerization 48.1 MPa (SD=7.5, n=8). Young's modulus of bending was 2089.3 MPa (SD=586.6, n=8) and 3781.5 MPa (SD=425.3, n=8) respectively for the same cement samples. Water sorption after two weeks for the autopolymerization cement samples was -1.12 wt% (SD=0.75, n=8) and for the light initiated polymerization samples 0.56 wt% (SD=0.20, n=8). Non parametric Spearman's correlation was measured for autopolymerized cement samples between variables for color and for solidification (load (N)), which showed a strong correlation between curing process and color change ($p<0.05$).

Conclusion. The results of this study showed that there was a correlation between the color change and degree of monomer conversion of the dual curing resin composite luting cement which contained a color indicator system for polymerization reaction. The study also suggested that autopolymerization only resulted in suboptimal polymerization of the cement. By additional light curing considerably higher flexural properties were obtained.

Keywords: color change, color indicator, resin cement, physical properties

Dual-curing resin cement with color indicator: Change of color by curing and some physical properties

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Declarations of interest: none.

1. Introduction

An effective adhesion of resin cements to dental tissues is foremost for the success of adhesively cemented restorations. However, the clinical success of these kinds of restorations is complex and exceeds a good adhesion between luting agent, restoration and abutment [1–3]. A number of factors add to the clinical performance of indirect restorations, including the type of resin cement, the activation mode of the polymerization, the photo polymerization time and the thickness of the luting cement [4–6]. Resin cements that are light-cured have been commonly used for the cementation of indirect ceramic and resin composite restorations due to their better time control during the cementation process. However, since the type of material and the thickness of the restoration can hamper the degree of monomer conversion by the polymerization reaction of the resin cement, a resin cement that does not depend entirely on light activation might be desired [7–9].

Resin cements can be classified based on their polymerization reaction, which takes place due to a light or chemical activation, in addition to the combination of the two processes that occurs in dual-cured cements. The bonding procedure is also used to categorize cements, including total etch, one-step etch bond and self-adhesive resins [10,11]. Chemically cured resin cements harden without the presence of light by combining activator and initiator components during mixing. However, the working time is not controlled by the clinician [12,13]. Differently, light-cured resin composite cements provide a convenient handling, but the thickness of indirect restorations might interfere the photopolymerization of the cement [14,15]. Dual-cured resin cements on the other hand reach adequate degree of cure and provide an extended working time due to the easily controlled light-curing mechanism. As a result, a fast initial setting of the resin cement contributes to a stabilization of the restoration [16,17]. A resin cement should be able to achieve a high degree of monomer conversion, which guarantees a more effective sealing ability, lower solubility of the cement, higher bond strength and superior color stability of the cement [18–20]. Dual-cured cements along with the benefit of favoring a better polymerization in deep areas of teeth, show elevated mechanical properties such as flexural strength and hardness when compared to those where other activation mechanisms are involved [21]. Interferences in the polymerization process can compromise the mechanical properties and clinical performance of the restorative material [22].

Removal of excess cement associated with indirect restorations is a crucial working step, and if done improperly excesses can be a risk factor of gingivitis, periodontitis, as well as perimplantitis in the case of implant supported restorations [23–25]. The resin cements may also adhere to the oral surface of restorations, which can be an inconvenience for the cleanup process and also add time to the cementation process. The removal of excess cement is important along with the determination of the correct moment of removal. The interruption of the light curing at an inappropriate time for removal of cement from non-desirable places has been investigated. Some affirm that this practice may result in a low cross-link density of the luting cement, translated into resin luting cement with higher susceptibility to wear [26,27]. Determining the appropriate time for cement removal can be difficult although material manufacturers offer time estimates. Some manufacturers offer a color cleanup indicator to identify the optimal time to remove excess cement.

To the authors knowledge there are no reports on visual indicators that state the precise time for excess cement removal in conjunction with the level of the degree of cure of resin composite luting cements. Therefore, the aim of this study was to investigate a dual-curing cement that provides a color cleanup indicator with relation of the color change to the degree of monomer conversion and to some mechanical properties of the cement.

2. Materials and methods

A self-etch, self-adhesive dual-cure resin cement was investigated (Maxcem Elite™ Chroma, Kerr, Orange, CA, USA). Clear color shade from the manufacturer's standard selection was used. A single operator prepared the specimens and performed the measurements. The cement was handled under ambient laboratory conditions ($23^{\circ}\text{C} \pm 1^{\circ}\text{C}$) using the normal lightning of the laboratory (630 lx at the working level). The cement was dispensed according to the manufacturer's instructions and inserted into a round shaped acrylic mold (1 mm thick and 4 mm in diameter) placed on a mylar strip.

The curing state of the autopolymerizing cement samples ($n=6$) was visually inspected with 1 minute intervals for 10 minutes and photographed. Color change of the cement during autopolymerization was further quantified by measuring spectrum of the reflected light using a spectrophotometer (CM-700d, Konica Minolta, Osaka, Japan). The color spectrum (CIELAB color space) was measured with 30 second intervals up to 10 minutes. Number of samples used for spectrophotometer measurement was 6 ($=n$).

The curing process (solidification) during autopolymerization of the cement was studied with a custom arrangement on a single column materials testing machine (Lloyd Instruments LRX, Ametek Inc, Largo, FL, USA) (Fig. 1). The cement was dosed into a 0.5 ml syringe which was cut 1 cm long and attached to a measuring table with two-sided adhesive tape. Next, a solid metal bar (diameter 1.2 mm) was pressed into the curing cement at constant speed of 0.3 mm/min ($n=6$). The force was limited to 50 N to prevent damage to the testing equipment and the results in Newtons were recorded as force over time which reflected autopolymerization of the cement. Force in Newton was used as an indicating unit for solidification, and further for plotting the state of autopolymerization to the color change.

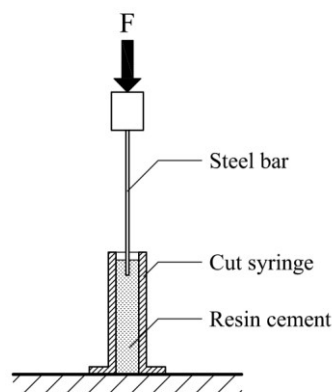


Figure 1. Custom test arrangement for measuring solidification of the cement during autopolymerization. Force in N was used as the indicating unit.

Additional specimens were made for measuring the flexural properties of the cement with a three-point bending test. The cement was dispensed into a $(2 \times 2 \times 25)\text{mm}^3$ mold, with a microscope glass on top. The samples were divided into two groups. The first group ($n=8$) was light cured for 20 seconds at a distance of 1 mm at each half of the specimen using a hand held light curing unit with reported light intensity of 1200 mW/cm^2 by manufacturer (Elipar™ S10, 3M Espe, St. Paul, MN, USA). The second

group (n=8) was let to autopolymerize for up to three minutes before taking the glass off. The specimens were then extracted from the molds, any excess material was removed, and were subsequently wrapped in aluminum foil and stored in a desiccator at 22°C for three days. Next, the samples were unwrapped and placed in water and stored at 37°C. Water sorption was measured by gently drying the specimens and placing them on a scale. The specimens were measured on days 1, 2, 4, 7 and 14 after placing them in water and the weight change (water sorption) was measured.

After the last weighing of the specimens was carried out, a three-point bending test was performed using a dual column universal testing machine (Lloyd Instruments LR30K, Ametek Inc, Largo, FL, USA). The span was set to 20 mm and a 2.5kN load cell was used. The results were recorded as maximum bending stress (MPa) over extension from preload (mm). In addition Young's modulus of bending was recorded for each sample.

The statistical analyses were performed using SPSS software (SPSS 21.0, IBM SPSS Statistics, Armonk, NY, USA) to determine Spearman's correlation between variables for color change and unit of solidification. In addition mean values of flexural strength and water sorption at the time point of 14 days were compared using an independent samples T-test.

3. Results

The values obtained for color variation, curing speed (time for solidification), water uptake and flexural properties are represented in Figures 1 to 6 accordingly. Results show the correlation between curing process and color change during the autopolymerization. Also, the final flexural strength results underline the importance of additional light curing to reach maximal material strength.

In figure 2 the photographed change of color from pink to final shade can be seen to happen mostly during time period of 2 to 4 minutes after mixing. Quantification of the color change was further measured by spectrophotometry which were in line with the visual findings by placing the time of most color change between 1 minute 30 seconds and 4 minutes after mixing. Results were measured in CIELAB color space, which uses one value (L) for luminance and two (a and b) for color. L is a value for lightness from black to white (0 to 100, respectively), a is a value for color from green to red (-128 to 127, respectively) and b is a value for color from blue to yellow (-128 to 127, respectively). The results are presented in figure 3.

In figure 4 the solidification of the cement (n=6) starts at around 160 seconds mark after mixing (2 minutes 40 seconds) and thus seems to align well with the color change. Solidification reaches steady rise from 230 seconds (3 minutes 50 seconds) mark onward up to the testing limit (force of 50 N).

Weight change in figure 5 was measured to be up to 0.56 wt% (SD=0.50, n=8) for light initiated polymerization samples after 14 days and -1.12 wt% (SD=0.75, n=8) for the autopolymerized samples. Negative values seem to suggest some material was dissolving during the test.

Finally the same samples used in water sorption were tested by the three-point bending. Results are shown in table 1 and representative load-deflection curves in figure 6. Results show that light initiated polymerization samples reached higher flexural strength in comparison with the autopolymerized samples.

Table 1. Results from three-point bending test for autopolymerized and light initiation polymerized cement samples.

Flexural strength (MPa)		
	Autopolymerization	Light initiation
Mean	27.34	48.06
S.D.	5.06	7.52

Young's modulus of bending (MPa)		
	Autopolymerization	Light initiation
Mean	2089.31	3781.51
S.D.	586.62	425.31

Non-parametric Spearman's correlation was measured between three variables – two for color (a and b values) and one for curing (load (N) value). For analysis time window of

the color change between 01:00 and 04:30 (minutes) was chosen. Curing measurement data was further formatted to fit the spectrometry measurement (30s intervals) by taking the mean value of 30s interval (for the 1 minute value the mean load from 30s to 1min was used).

The results show strong correlation between curing and color change with the correlation between the variable A and load varying from -1.000 to -0.833, and between the variable B and load varying from 0.833 to 1.000. The results were statistically significant ($p < 0.05$).

Additionally, means of the final flexural strength and water sorption between autopolymerized and light initiation polymerized groups ($n=8$) were compared using an independent samples T-test. Analysis showed statistically significant difference between the groups ($p < 0.05$) in both categories.

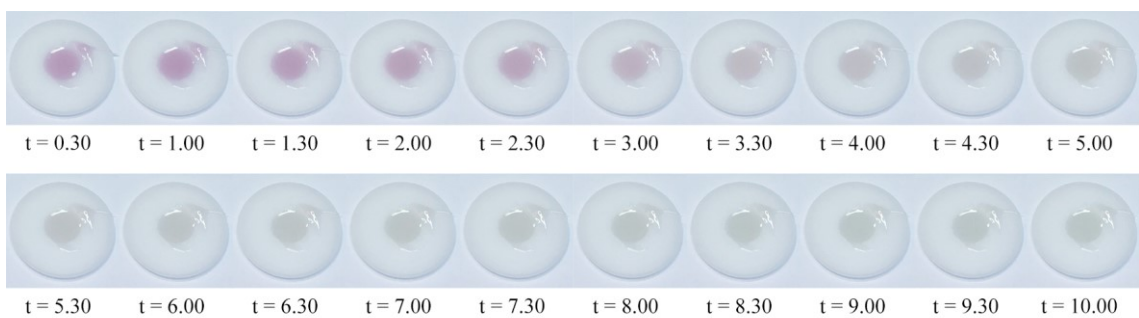


Figure 2. Photographs of the color change of the cement during autopolymerization (“t” refers to time in minutes after the start of the autopolymerization).

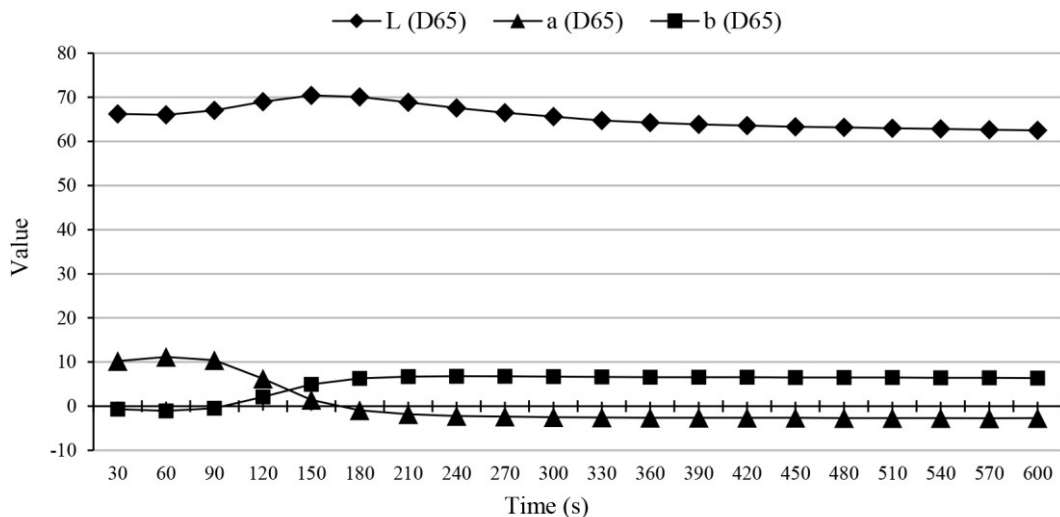


Figure 3. Spectrometry values of the color change of the cement during autopolymerization. Results in CIELAB color space where L is the value for lightness (0 to 100), a is the value for color from green to red (-128 to 127) and b is the value for color from blue to yellow (-128 to 127).

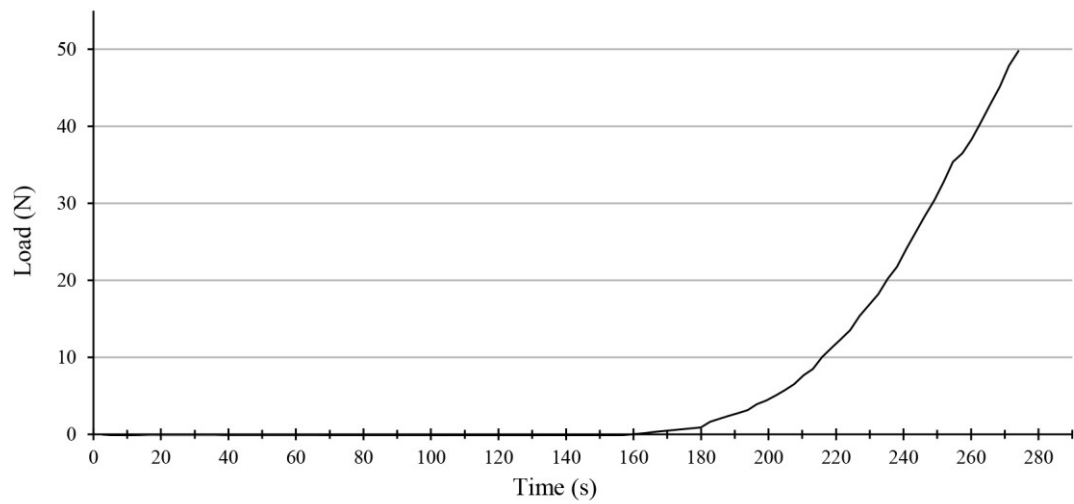


Figure 4. Solidification profile of the cement during autopolymerizing.

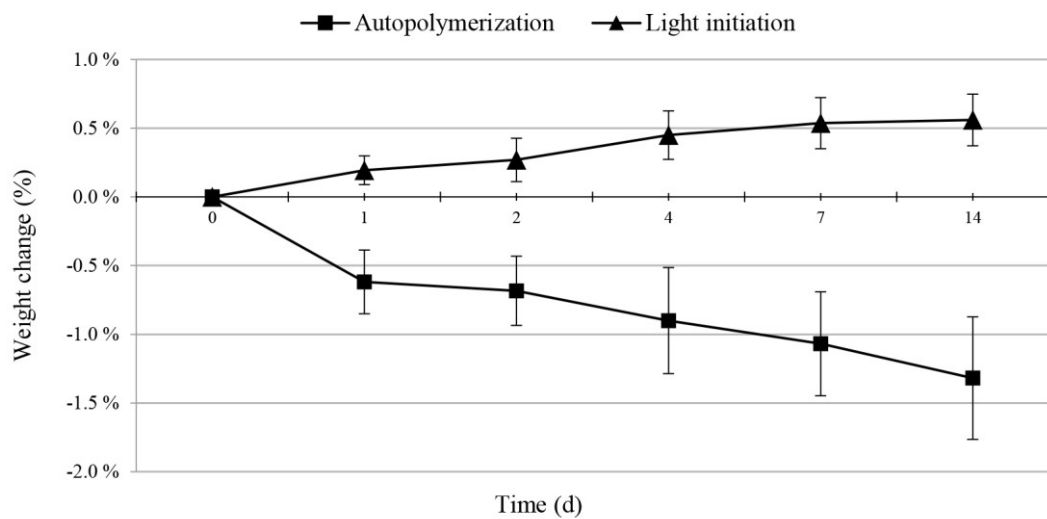


Figure 5. Weight change in water of samples after the light initiated and autopolymerized cement samples after 1/2/4/7/14 days.

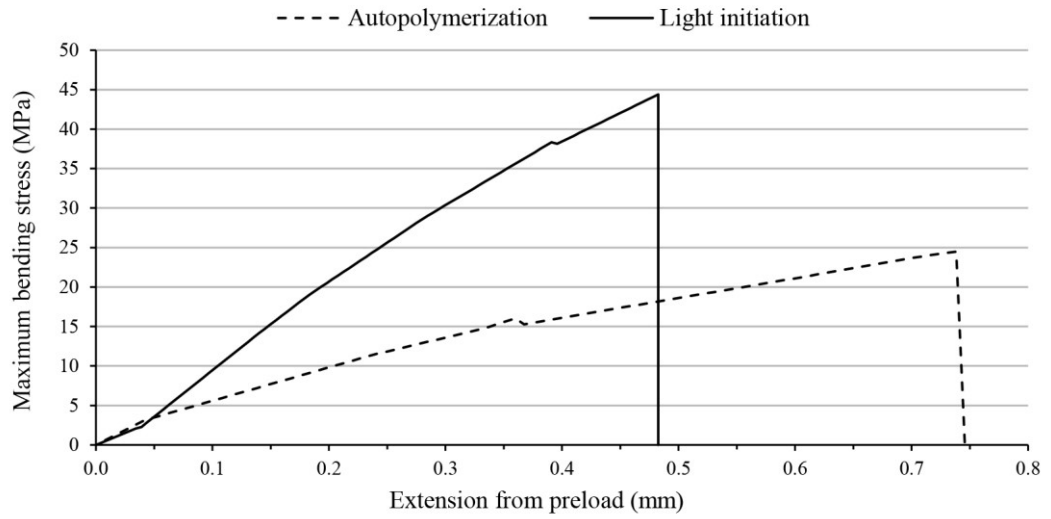


Figure 6. Representative load-deflection curves for test specimens which were polymerized by light initiation and autopolymerization.

4. Discussion

The aim of this study was to investigate the color change of a self-adhesive, dual-cured cement that provides a color cleanup indicator (Maxcem Elite™ Chroma, Kerr, Orange, CA, USA) and to evaluate its mechanical properties. A correlation between the color variation in the cement and its mechanical properties was found. The color fading indicated the optimal window of time to clean up excess cement.

The material investigated is available in a dual barrel, dosed through automix mixing tips and is of pink color when applied. The color then gradually changes into the final color shade, which occurs during the first minutes after mixing, when the cement is let to autopolymerize. After the pink color has faded any excess material is safe to be removed, according to manufacturer. The final bond strength to tooth substance and restoration is then achieved by light curing. The manufacturer also states that it is possible to use tack curing method to speed up the excess removal. When tack curing, cement is light cured for 1-2 seconds and then excess material is removed. The final curing is done according to the manufacturer's specifications. Tack curing method was not used in this study as the color change is near instant with light curing. As the initial visual inspection, digital photography and further testing of the autopolymerization process showed, the fading of most of the original pink color took place during the first 2-3 minutes, by which time the cement had started to cure and was already transformed from a gel state into a more stiffer form.

Although the exact substances or mechanics causing the color change of the studied cement are not known, this study was undertaken to get more in-depth data on the nature of the color change and its relation to curing. In the field of polymer sciences, it has been shown, that the use of commercially available dyes and certain other reagents can result in a color change during polymerization [28,29]. For example, the use of lead dithizone or carbazone in poly(methylmethacrylate) (PMMA) results in a color change from red to yellow during the polymerization [28]. When lead dithizone is present, the PMMA polymerization proceeds by a free radical mechanism and the color change is attributed to formation of azo group by lead dithizone (affected by free radicals) [28]. When the polymerization proceeds and all of lead dithizone has reacted, the final color is achieved. The use of bromophenol blue with trimethylene glycol dimethacrylate (TGDM) has also been recorded [29]. In this case the polymerization also proceeds by a free radical mechanism and the dye loses its color when reacting with the free radicals. By adjusting the amount of dye in the final product it could be possible to time the color change with a certain curing level. While bromophenol blue interacts via free radicals, it is worth mentioning that it also acts as a pH indicator, thus changing its color via a different reaction. As some resin cements exhibit a pH change when curing [30] it might also be possible to use such common pH indicator as pentamethoxy red to determine the degree of cure.

The curing process, i.e. the solidification of the autopolymerizing cement was studied in a custom testing arrangement. The used method was chosen by reason of that spectrometry was not an option due to the self-etching properties of the cement, and thus a potential damage to the infrared sensor would be involved. The testing arrangement provided results of the time needed for solidification of the cement and showed a slowly speeding up curing from around 160 seconds after mixing. The curing speed then started to accelerate and reached a steady rise after 230 seconds. The curing

speed and timing was in relation to the color change happening around 2-3 minutes mark as shown in the spectrometry and visual examination.

The weight change (water uptake) of the light cured samples was measured to be 0.56 wt% after 14 days but the autopolymerized samples resulted in a negative value of -1.12 wt% during the same period of time. This would suggest that there are compounds leaching from the non-light cured samples. It is likely that major releasing compounds are residual monomers. More detailed analysis of the leaching compounds could have been performed by high performance or gas chromatography and there are interests by the authors for studying this aspect in the future studies. The three-point bending test showed a clear difference in flexural strength in favor of the light cured samples. Autopolymerized samples did show higher extension from preload before breaking but with considerably lower flexural strength. Findings are in line with the fact that composite cements such as the one under study here achieve higher degree of conversion, even if light cured through ceramic material [31,32]. Thus, it is suggested that light curing is required for optimizing curing of the cements and clinical outcome of indirect restorations cemented with the studied luting cement.

5. Conclusion

The results of this study showed that there was a correlation between the color change and state of curing of the dual curing resin composite luting cement which contained a color indicator system for polymerization reaction. The study also suggested that autopolymerization only was not adequate for the optimal polymerization of the cement. By light curing considerably higher flexural strength was obtained.

6. Acknowledgments

The authors extend their appreciation to the International Scientific Partnership Program ISPP at King Saud University for funding this research work through ISPP#0088. The authors thank DDS Lippo Lassila and M.Po.Sc Jouko Katajisto for their support.

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